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**Petroleum evolution within the Tarim Basin, NW China: Insights from organic geochemistry, fluid inclusions, and Re-Os geochronology of the Halahatang Oilfield**

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33

## Abstract

The newly discovered Halahatang oilfield in the northern Tarim Basin has a potential resource of > 70 Bbbls of oil. Oil organic geochemical data from the Halahatang oilfield indicate that the oils are of moderate maturity, biodegraded, and represent one oil family, derived from the same Paleozoic marine source. Modeling of coeval aqueous and hydrocarbon-bearing inclusion data provide fluid trapping temperatures and pressures of 100 to 110 °C and ~39 to 59 MPa, respectively. The fluid inclusion data coupled with the previous basin model studies, suggests a single prolonged oil migration event during the Permian. Rhenium-Osmium (Re-Os) isotope data oil yield an Early Permian Re-Os age of  $285 \pm 48$  Ma. The age agrees with the timing of maturation of the Paleozoic source via burial history modelling but is slightly older (~5 - 55 myr) than the oil migration/accumulation timing implied by the basin modelling coupled with fluid inclusion analysis and the published reservoir illite K-Ar dates. Thus, the oil Re-Os date suggests that oil generation in the Halahatang Depression of the Tarim Basin occurred during the Early Permian, rather than the Silurian as previously proposed, with subsequent oil migration/accumulation occurring during the Mid-Late Permian as recorded by basin modelling, coupled with fluid inclusion analysis and illite K-Ar dating. In addition to promoting petroleum exploration in the Tarim Basin, this study that combines crude oil Re-Os isotope dating and traditional analytical methods (organic geochemistry/fluid inclusion analysis) to constrain petroleum evolution is applicable to hydrocarbon systems worldwide.

## Keywords

Petroleum evolution; Re-Os geochronology; Organic geochemistry; Fluid inclusions; Halahatang oilfield; Tarim Basin

## 1 Introduction

The accurate key timing of petroleum evolution (e.g., oil generation, migration / accumulation) is vital to understand the evolution of a petroleum system (Liu et al., 2013; Qiu et al., 2011), and is crucial for hydrocarbon exploration in a target region (Roberts et al., 2004). Although thousands of oilfields are known worldwide, understanding how to precisely constrain the key events of a petroleum system remains challenging (Liu et al., 2013; Mark et al., 2010; Roberts et al., 2004). Aimed at solving these problems, the oilfield in Tarim Basin, northwest China was selected as an example in this study. The Tarim basin is bordered by the Tian Shan, West Kunlun and A'erjin orogenic belts to the north, southwest, and southeast, respectively (Lin et al., 2015; Zhang et al., 2007a) (Fig. 1B). The basin encompasses an area of 560,000 km<sup>2</sup> (~216,217 mi<sup>2</sup>) and contains up to ~14 km (~8.7 mi) of sedimentary strata. It has had a complex tectonic evolution (e.g., Caledonian, Hercynian, Indosinian-Yanshan and Himalayan orogenies). The basin contains significant petroleum potential (more than 70 billion barrels oil (Bbbls) and 250 trillion cubic feet (Tcf) of gas) (Lin et al., 2015; Xiao and Tang, 2003; Xu et al., 2004). The Tarim Basin is the largest known onshore petroliferous basin in China, with only an estimated 10 percent of the total potential reserves presently discovered (Li, 2009; Xu et al., 2004). Although the Tarim Basin is considered to contain the most important future oil and gas resources in China (Li, 2009), the multiple tectonic events that the basin

records have led to a complex hydrocarbon evolution, which has hampered oil and gas exploration (Li, 2009; Lin et al., 2015; Zhang et al., 2007a).

More than thirty oil fields (e.g., Dawanqi, Lunnan, Tahe, Tazhong, Hetian and Bashituo) have been discovered throughout the Tarim Basin during the past three decades (Xu et al., 2004; Zhao et al., 2004). More recently discovered is the deeply buried (>6000 m) (>19,685 ft) Early Paleozoic carbonate Yijianfang Formation in the Halahatang depression of the northern Tarim Basin that has a current estimated reserve of >4 Bbbls of oil (Zhu et al., 2013a), which supports the reported resource potential within the Tarim Basin (Li, 2009; Xu et al., 2004). However, both the source and timing of hydrocarbon generation and accumulation in the Tarim Basin remain debated (Chang et al., 2013a; Li, 2009; Li et al., 2010; Liao et al., 2010; Tian, 2005; Xiao et al., 2012). Hydrocarbon maturation models propose that the Neoproterozoic and Early Cambrian shales are the main source for the Shaya Uplift oilfield in the northern Tarim Basin (Li, 2009) and the Late Cambrian to Ordovician shales are the suggested oil source in Kongquehe area in northeast Tarim Basin (Tian, 2005). Based on both biomarker and  $\delta^{13}\text{C}$  analysis of individual *n*-alkanes for oils, a mixed origin (Cambrian–Lower Ordovician and Middle–Upper Ordovician) has been proposed for the central Tarim Basin (Li et al., 2010). Basin modeling in the Caohu Depression in the northern Tarim Basin considers the Early Ordovician (ca. 400 Ma) to be a key time of oil generation and migration (Tian, 2005). This is, in part, supported by authigenic illite K-Ar dating (ca. 380 Ma) from the Late Silurian sandstone reservoir in the central Tarim Basin (Zhang et al., 2004). However, younger migration ages are also proposed in the central and northern Tarim Basin by authigenic illite K-Ar dates of ca. 250 and ca. 20 Ma (Zhang et al., 2004; Zhu et al.,

103 2013c).

104 In the newly discovered Halahatang oil field, previous GC-MS (terpane and sterane  
105 characteristics) analysis on the oil and potential source rocks invoke both the shales of the  
106 Cambrian Yuertusi Formation, as well as Middle-Late Ordovician carbonates of the  
107 Lianglitage and Sangtamu Formations as the main sources of the petroleum (Chang et al.,  
108 2013a; Lu et al., 2008; Xiao et al., 2016; Zhu et al., 2013a). The current burial history  
109 models and fluid inclusion data propose several age models for the petroleum evolution  
110 in the Halahatang oil field (e.g., Late Silurian, Late Permian and Neogene) (Chang et al.,  
111 2013a; Si, 2013; Xiao et al., 2012; Zhu et al., 2013a). As a result the following key  
112 aspects of the petroleum systems are debated: (1) the source(s) of the oil; (2) the timing  
113 of oil generation, and (3) the timing of oil charging within the Tarim Basin.

114 Rhenium-osmium (Re-Os) isotope analysis on hydrocarbons has shown potential to  
115 determine the absolute timing of hydrocarbon generation (Cumming et al., 2014; Finlay  
116 et al., 2011; Ge et al., 2016; Ge et al., 2018a; Ge et al., 2018b; Georgiev et al., 2016;  
117 Lillis and Selby, 2013; Selby and Creaser, 2005; Selby et al., 2007). Further, authigenic  
118 illite K-Ar (Ar-Ar) dating from the oil reservoir and fluid inclusion studies can help  
119 record the timing of oil migration and reservoir filling (Guo et al., 2012; Hamilton et al.,  
120 1989; Lee et al., 1985; Mark et al., 2010; Zhang et al., 2004). In this study, we apply and  
121 discuss new oil geochemical analysis, fluid inclusion analysis, and Re-Os geochronology  
122 along with previous published sandstone authigenic illite K-Ar dating (Zhang and Luo,  
123 2011; Zhu et al., 2012), to quantitatively determine the petroleum evolution (timing of oil  
124 generation, migration/accumulation) associated with the Halahatang oilfield of the  
125 northern Tarim Basin. In addition, this work demonstrate this combined approach can

yield quantitative data to establish the timing for petroleum evolution that may aid in the further understanding of both the temporal and spatial evolution of hydrocarbon systems worldwide.

## **2 Geological setting**

The Halahatang depression occupies an area of ~40,000 km<sup>2</sup> (15,444 mi<sup>2</sup>) within the centre of the Tabei Uplift in the northern Tarim Basin (Zhu et al., 2011). The Halahatang depression is bordered by the Yingmaili, Luntai, and Lunnan Uplift to the west, north and east, respectively, and the North Depression to the south (Fig. 1C). The geology of the Halahatang depression is characterized by a thick sequence of Cambrian to Quaternary strata (~14 km) (~8.7 mi) (Fig. 2) (Jia and Wei, 2002; Zhang and Huang, 2005). The Cambrian to Ordovician strata (~4.5 km) (~2.8 mi) comprise shallow marine to lagoonal carbonates (Jia and Wei, 2002), with the Silurian to Devonian strata being represented by ~1 km (~0.62 mi) of fine-grained red beds and sandstones (Zhang and Huang, 2005), and the Carboniferous to Permian section is characterized by a ~1 km (~0.62 mi) thick interval of sandstone and mudstone (Chang et al., 2013b). Since the Triassic the renewed subsidence of the Halahatang depression has led to the accumulation of ~6 km (3.73 mi) of Mesozoic to Cenozoic fluvio-lacustrine sediments (Zhang and Huang, 2005).

The Halahatang depression records multiple tectonic events. The Late Ordovician Caledonian Orogeny resulted in the uplift of the northern and central parts of the Tarim Basin (Zhang et al., 2007a). The region encompassing the Halahatang depression existed as a marginal foreland basin until the Late Permian (Jia and Wei, 2002; Wei et al., 2000; Zhu et al., 2011), and suffered uplift during the Hercynian Orogeny (Jia and Wei, 2002).



Since the Triassic the Halahatang depression experienced several burial and uplift events controlled by the closure of the Tethys Ocean and collision between Indian and Eurasian Plates (Yanshan and Himalayan orogenies) (Jia and Wei, 2002; Xu et al., 2016; Zhang and Huang, 2005; Zhu et al., 2011).

In the Halahatang depression, the carbonates of the Middle Ordovician Yijianfang Formation (>6,000 m deep) (>19,685 ft deep) is the main hydrocarbon reservoir, with Silurian and Triassic sandstones also considered as potential reservoir units (Zhu et al., 2013a) (Fig. 2). The source rock for the oil is still debated, but the main sources are considered to be the shales of the Cambrian Yuertusi Formation, and organic-rich carbonates of the Middle to Late Ordovician Lianglitage and Sangtamu formations (Chang et al., 2013b; Cui et al., 2009; Huo et al., 2016; Xiao et al., 2016). The dense shale or mudstone above the reservoirs are considered the cap rocks (Zhang and Huang, 2005; Zhu et al., 2013a).

### 3 Samples and methods

Five oil samples from separate wells (Ha9, Ha11, Ha15-2, Ha701 and XK4-3) in the Halahatang depression were collected for GC-MS and Re-Os analysis (Fig. 1C). All the oil samples are from the Middle Ordovician Yijianfang Formation between ~6550m (21,489 ft) and 6850 m (22,474 ft) (Table 1). The oil physical property and organic compositions were collected from the unpublished reports of the Tarim Oil and Gas Company. The experiments were conducted at Research Institute of Petroleum Exploration and Development, PetroChina, Beijing with the oil density, oil viscosity and oil fractions measured following three different national standard methods (General

Administration of Quality Supervision Inspection and Quarantine of the People's Republic of China, 2011; National Development and Reform Commission, 2008; National Energy Administration of the People's Republic of China, 1993). The oil density, viscosity, and API values are 0.83 - 0.10, 3.15 - 342.3, and 8.46 - 38.37, respectively. The organic compositions, wax, sulfur, saturate, aromatic, resin, and asphaltene of the oil are ~6.0 %, ~0.6 %, ~56.1 %, ~16.5 %, ~4.7 %, and ~18.7 %, respectively. All the oil samples, with the exception of the oil from Well Ha701, are characterized by low viscosity (<0.90), a high API value (>29), and low asphaltene content (<25 %) (Table 1). Thus all the oils are characterized as light crude oil. However, the oil from well Ha701 possesses a high viscosity (~342) and asphaltene content (~34 %) and low API value (8.46) (Table 1), which characterizes the oil as heavy oil (Schenk et al., 2006; Zhang et al., 1990).

The gas chromatography mass spectrometry (GC-MS) analysis of the oil samples were conducted at the China University of Geoscience (Wuhan) following the analytical procedure of (Zhang et al., 2015). Approximately 30 mg of crude oil sample was dissolved in 50 ml of *n*-hexane and left for 12 hrs at room temperature. The solution was then filtered, with all the filtrates collected and evaporated under nitrogen gas to 0.5 ml. A chromatographic column (30 cm × 10 mm in diameter) was prepared using a mixed stationary phase of activated silica gel and alumina with a ratio of 3:2 (Yang et al., 2009).

The concentrated sample was transferred to the chromatographic column for further separation. The saturated hydrocarbon fraction was eluted with *n*-hexane (25 ml). The fractions were then carefully concentrated under nitrogen flow to 0.5 ml with the concentration around 5-10 mg/ml for GC-MS analysis. The GC-MS system consists of

195 the Agilent 7890 GC and 5975C mass spectrometers. A DB-5MS column 50 m × 0.25  
196 mm × 0.25 µm was used. High purity helium (99.9995 %) was used as a carrier gas with  
197 a flow rate of 1.0 ml/min. The injector temperature was 300 °C. The injection volume  
198 was 1.0 µl. All injections were done with a 7683B series autosampler. The oven  
199 temperature was programmed from 50 °C (1 min hold) to 100°C at 10°C /min, and then  
200 to 310°C (20 min hold) at 2°C /min. The mass spectrometer was operated in the electron  
201 impact mode (70 eV). The temperature of ion source and transfer-line were set at 230°C  
202 and 300°C, respectively. The scanned mass range was from 50 to 550 u. The temperature  
203 of the quadrupole was held at 150°C.

204 For the oil Re-Os analysis, the asphaltene fraction was analyzed as Re and Os are  
205 predominantly contained within the asphaltene fraction of oil (Cumming et al., 2014;  
206 Georgiev et al., 2016; Lillis and Selby, 2013; Rooney et al., 2012; Selby et al., 2007). The  
207 asphaltene fraction was precipitated from the oil using 40 times volume of *n*-heptane  
208 (~1g oil with 40 ml solvent) at room temperature for at least 8 hrs. The Re and Os  
209 isotopic compositions and abundances of the asphaltene from the oil were analysed at the  
210 Laboratory for Source Rock and Sulfide Geochronology and Geochemistry and the  
211 Arthur Holmes Laboratory (members of the Durham Geochemistry Centre) at Durham  
212 University following published analytical procedures (Selby et al., 2005; Selby et al.,  
213 2007). Approximately 100-200 mg of asphaltene were dissolved and equilibrated with a  
214 known amount of a mixed <sup>185</sup>Re and <sup>190</sup>Os spike solution by inverse *aqua-regia* (3 ml  
215 HCl and 6 ml HNO<sub>3</sub>) in a Carius tube for 24 hours at 220°C. Osmium was isolated and  
216 purified from the inverse *aqua-regia* by CHCl<sub>3</sub> solvent extraction at room temperature  
217 and micro-distillation. The Re was isolated using HCl-HNO<sub>3</sub>-based anion

chromatography. The purified Re and Os were loaded on Ni and Pt filaments, respectively, and analyzed using negative ion thermal ionization mass spectrometry (NTIMS). Total procedural blanks for Re and Os are  $1.60 \pm 0.03$  pg and  $0.05 \pm 0.01$  pg, respectively, with an average  $^{187}\text{Os}/^{188}\text{Os}$  ratio of  $0.22 \pm 0.06$  (1 SD;  $n = 4$ ). All uncertainties include the propagated uncertainty in sample and tracer solution weights, the standard, spike calibrations, mass spectrometry measurements, and blanks. In-house Re (Restd) and Os (DROsS) solutions were analyzed as a monitor of reproducibility of isotope measurements. The  $^{187}\text{Os}/^{188}\text{Os}$  values of the Os standard solution DROsS during this study are  $0.1611 \pm 0.0004$  (1SD,  $n = 126$ ), with the  $^{185}\text{Re}/^{187}\text{Re}$  values of the Re standard solution being  $0.5989 \pm 0.0019$  (1SD,  $n = 116$ ). These values are in agreement with those previously published for DROsS and Restd (Cumming et al., 2014; Finlay et al., 2011, 2012; Lillis and Selby, 2013; Nowell et al., 2008). The Re–Os data of this study are regressed using the program *Isoplot* V. 4.15 (Ludwig, 2003) with  $^{187}\text{Re}$  decay constant of  $1.666 \times 10^{-11} \text{ a}^{-1}$  (Smoliar et al., 1996). The input data contains  $^{187}\text{Re}/^{188}\text{Os}$  and  $^{187}\text{Os}/^{188}\text{Os}$  ratios with their total absolute  $2\sigma$  level uncertainty and the associated error correlation, Rho (Ludwig, 1980).

A doubly polished fluid inclusion wafer (~100 micron thick) of a bioclastic limestone from the Ordovician Yijianfang Formation from well Ha9 was prepared for the fluid inclusion studies (Fig 3). Fluid inclusion petrography, microthermometry, laser raman microspectroscopy (LRM) and confocal scanning laser microscopy (CSLM) were conducted by CV Associés Engineering, Nancy, France.

Fluid inclusion petrography was carried out using a Zeiss Axiovert 200 microscope equipped with both transmitted white and incident ultraviolet light (UV) ( $\lambda = 365$  nm). A

calibrated Linkam MDS 600 heating and cooling stage was used for microthermometry. Homogenization temperatures ( $T_h$ ) were obtained using the thermal cycling method with a heating rate of 10 °C/min (Goldstein and Reynolds, 1994). The measured temperature precision for the  $T_h$  is  $\pm 0.1$  °C. The LRM analyses of aqueous fluid inclusions were performed on a Labram Jobin Yvon spectrometer, using 514.5 nm radiation produced by an argon laser. The salinity of aqueous fluid was estimated by LRM following the method described by (Dubessy et al., 2002). The CSLM methodology was used to measure the gas/oil volume ratio ( $F_v$ ) of the hydrocarbon inclusions previously identified by UV-fluorescence and characterized by microthermometry. The measurements were carried out using a Bio-Rad (Zeiss) Radiance 2100 Rainbow confocal scanning laser microscope equipped with an argon laser emitting at 488 nm and mounted on a Nikon TE2000-U inverted microscope.

## 4 Results

### 4.1 GC-MS analysis

Abundant biomarkers (e.g., alkane and isoprenoids, terpane and steroids) were detected in all five oil samples (Table 2). The saturate fraction gas chromatograms (SFGCs) for three of the oils (Ha9, Ha701, XK4-3) exhibit an unresolved complex mixture (UCM) (Fig. 4), however, abundant alkane compounds are still detected in the oil samples above the UCM (Fig. 4). The carbon number of the alkane distributed between  $nC_{12}$  and  $nC_{27}$ , with the highest peak occurring at  $nC_{15}$  or  $nC_{16}$  (Fig. 4). For the isoprenoids, the Pristane (Pr)/Phytane(Ph) ratios of the five oil samples range between 0.68 and 0.97. The ratios of Pr/ $nC_{17}$  and Ph/ $nC_{18}$  range from 0.04 - 0.47 and 0.07- 0.64, respectively (Table 2)(Fig.

5A, B). Tricyclic terpanes, tetracyclic terpanes and hopanes were detected in the oil samples ( $m/z$  191) (Fig. 4). The tricyclic terpanes range from  $C_{19}$  to  $C_{30}$ , with a clear abundance increase between  $C_{20}$ ,  $C_{21}$ , and  $C_{23}$  compounds (Fig. 4). The  $C_{24}$  tetracyclic terpane ( $C_{24}$ TET) is detected in the oils. The ratios of  $C_{19}/C_{23}$  tricyclic terpane ( $C_{19}$ TT/ $C_{23}$ TT), and  $C_{24}$  tetracyclic /  $C_{26}$  tricyclics terpane ( $C_{24}$ TET/  $C_{26}$ TT) are respectively 0.12 to 0.20 and 0.42 to 0.49 (Table 2)(Fig. 5C). The hopanes range from  $C_{27}$  to  $C_{35}$  and exhibit the highest peaks at either  $C_{29}$  or  $C_{30}$ . The hopanes show a decrease in the abundance with increasing carbon number between  $C_{31}$  and  $C_{35}$  (Fig. 4). Additional compounds (e.g.,  $C_{30}$  diahopane ( $C_{30}$ DH), Ts (18 $\alpha$ (H)-trishnorhopane), Tm (17 $\alpha$ (H)-trishnorhopane), gammacerane and 25-nor-hopane) are also detected. The Ts/(Ts+Tm) and  $C_{30}$ DH/ $C_{30}$ H ( $C_{30}$  hopane) ratios vary from 0.37 to 0.55 (except for Ha15-2 which has a value of ~0.03) and 0.03 to 0.13, respectively (Table 2)(Fig. 5D). The gammacerane/ $C_{30}$ H ratio varies from 0.05 to 0.19, with an average of 0.12, and the 25-nor-hopane/ $C_{30}$ H ratios range from 0.15 to 2.61 (Table 2)(Fig. 5F). Sterane compounds, such as  $C_{21}$  pregnane ( $C_{21}$ P),  $C_{22}$  homopregnane ( $C_{22}$ HP), diasterane, and  $C_{27}$ - $C_{29}$  sterane are detected (Fig. 4). The ratio of pregnane/homopregnane ( $C_{21}$ P/ $C_{22}$ HP) ranges from 2.98 to 5.69, with an average of 4.03. The  $C_{27}$ ,  $C_{28}$ ,  $C_{29}$  sterane content of all the oil samples present a similar V-shape distribution, which display ~50.2, 14.6 and 35.1 %, respectively, with  $C_{27}$  sterane exhibiting the greatest abundance. The ratio of  $C_{29}\alpha\alpha\alpha$  20S/(20S+20R) and  $C_{29}\beta\beta/(\beta\beta+\alpha\alpha)$  vary from 0.30 to 0.48 and 0.55 to 0.58 (Fig. 5E).

## 4.2 Fluid inclusion analysis

The fluid inclusion wafer from well Ha9 is composed of grains (mainly ooids, echinoderms and mollusks) enclosed by micrite and coarse calcite cements. Fluid

inclusions in both the cement and calcite replaced grains were studied. The majority of the hydrocarbon and aqueous inclusions are distributed along annealed microfractures both in the calcite cements and the calcite replaced grains. Hydrocarbon-bearing inclusions (typically  $\leq 5\mu\text{m}$  wide and  $\sim 2$  to  $10\mu\text{m}$  long; Fig. 3) are liquid-rich, two-phase (L+V; L>V) inclusions that display blue, green and yellow UV fluorescence and are brown in transmitted light. Some localized inclusions within the same crystal possess highly variable liquid/vapor ratio indicating late alteration events (leakage and/or necking down) (Table 3).

The majority of the aqueous inclusions are two-phase liquid rich inclusions (L+V; L>V) (Fig. 3). Rare monophasic liquid-rich aqueous inclusions were also encountered. All the fluid inclusions are  $\leq 5\mu\text{m}$  wide and  $\sim 2$  to  $15\mu\text{m}$  long. Both aqueous and hydrocarbon inclusions commonly occur along the same annealed microfractures indicating a coeval relationship between the fluids.

The homogenization temperatures ( $T_h$ ) values of the hydrocarbon-bearing inclusions range from  $24.6$  to  $122^\circ\text{C}$  (Table 3), with the majority homogenizing between  $24.6$  and  $79.9^\circ\text{C}$  (Fig. 6A). The aqueous inclusions homogenize between  $61.2$  and  $141.0^\circ\text{C}$  (Fig. 6A). The aqueous inclusions that are coeval with the hydrocarbon-bearing inclusions homogenize between  $61.2$  and  $102.3^\circ\text{C}$ , with a mean  $T_h$  of  $82.1^\circ\text{C}$  (Fig. 6A). The calculated salinities obtained using the method of Dubessy et al., 2002 vary from  $6.7$  to  $20.4$  wt. % NaCl eq. (Table 3), with the majority yielding values between  $12$  and  $16$  wt. % NaCl eq. The coeval hydrocarbon and aqueous inclusions have salinity values between  $8.6$  and  $15.1$  wt. % NaCl eq (Fig. 6B).

The gas/oil volume ratio ( $F_v$ ) measured by confocal scanning laser microscopy (CSLM)

of three hydrocarbon-bearing inclusions, (ranging in size between 22 and 302  $\mu\text{m}^3$ ), ranges from 5.1 to 10.9 %. The bubble and bulk volumes of the hydrocarbon inclusions range between 1.8 and 5.6  $\mu\text{m}^3$ , and 22.0 and 84.4  $\mu\text{m}^3$  (Table 3). In general, a positive relationship exists between the Th and Fv data (Fig. 6C).

LRM analysis was conducted on 14 aqueous inclusions. One coeval aqueous inclusion with a Th of 83.2 °C (similar to the mean data, 82.1 °C) was characterized and used for the estimation of trapping conditions of both aqueous and hydrocarbon fluids (Table 3, Fig. 7C). The CH<sub>4</sub> content and salinity measurement were performed on 11 aqueous inclusions in calcite. Only two samples possess CH<sub>4</sub> above the detection limit (0.017 and 0.075 molal) (Table 3). Both aqueous inclusion thermodynamic (AIT) modeling using the CH<sub>4</sub>-H<sub>2</sub>O-NaCl system (Duan et al., 1992; Guillaume et al., 2003), and hydrocarbon bearing fluid inclusion thermodynamic (PIT) modeling were used to estimate aqueous and hydrocarbon fluid trapping conditions (Montel, 1993; Pironon, 2004) in well Ha9 in the Halahatang oilfield (Fig. 7C).

#### 4.3 Re-Os analysis

The asphaltene Re and Os abundances of the five oil samples vary between 0.06 and 9.47 ppb, and 4.9 and 57.2 ppt, respectively (Table 4). Both the Re and Os abundances of some of the oil samples are lower than those previously reported for oil and bitumen from both hydrocarbon or metalliferous systems (Cumming et al., 2014; Finlay et al., 2011; Ge et al., 2016; Georgiev et al., 2016; Lillis and Selby, 2013; Selby et al., 2005), with most of the samples possessing higher Re and Os abundances when compared with that of the average upper crust (Re: 0.198 ppb and Os: 31 ppt) (Esser and Turekian, 1993; Rudnick and Gao, 2003).



The  $^{187}\text{Re}/^{188}\text{Os}$  values of the oil range from ~78 to 1655 and exhibit a radiogenic  $^{187}\text{Os}/^{188}\text{Os}$  composition of 1.48 to 4.68 (Table 4). Repeat analysis of oil samples Ha9 and Ha15-2, using a separately isolated asphaltene fraction, yield very reproducible  $^{187}\text{Re}/^{188}\text{Os}$  (125.4 vs 125.2 and 1655.2 vs 1636.7) and  $^{187}\text{Os}/^{188}\text{Os}$  (1.66 vs 1.74 and 2.25 vs 2.25) values, and similar Re (0.81 vs 1.23 ppb and 9.47 vs 5.86 ppb) and Os (37.4 vs 57.2 ppt and 35.2 vs 22.0 ppt) abundances (Table 4). Similar reproducibility has also been shown by previous studies (Lillis and Selby, 2013; Liu et al., 2018; Selby et al., 2005).

Collectively all the Re-Os data of all five oil samples do not yield any meaningful date (Fig. 8) as sample Ha15-2 possesses a distinct Re-Os isotope composition to the remaining four oil samples that display a positive correlation between  $^{187}\text{Re}/^{188}\text{Os}$  and  $^{187}\text{Os}/^{188}\text{Os}$  (Fig. 8). The Re-Os data, without sample Ha15-2, yield a Model 3 (assumes that the scatter in the degree of fit of the data is a combination of the assigned uncertainties, plus a normally distributed variation in the  $^{187}\text{Os}/^{188}\text{Os}$  values (Ludwig, 2008) date of  $285 \pm 48$  Ma ( $n = 5$ , MSWD = 6.1), with an initial  $^{187}\text{Os}/^{188}\text{Os}$  composition of  $1.08 \pm 0.20$  (Fig. 8).

## 5 Discussion

### 5.1 Oil Geochemistry of the Halahatang Oilfield

The biomarker molecular composition of an oil (*n*-alkanes, terpane, sterane) records information about its origin, maturity and alteration (Peters and Moldowan, 1993b; Wu et al., 2012; Zumberge, 1987). The carbon number of the alkane distributed between  $n\text{C}_{12}$  and  $n\text{C}_{27}$ , with the highest peak occurring at  $n\text{C}_{15}$  or  $n\text{C}_{16}$  (Fig. 4), indicating the oil is

mainly sourced from bacteria and algae (Peters et al., 2005). The ratios of  $\text{Pr}/n\text{C}_{17}$  and  $\text{Ph}/n\text{C}_{18}$  range from 0.04 - 0.47 and 0.07- 0.64, respectively (Table 2) (Fig. 5A) and indicate the oils are from marine or saline facies sourced from lower bacteria and algae (Zhang et al., 2011). The pristane/phytane ( $\text{Pr}/\text{Ph}$ ) ratio is a useful parameter to establish the depositional environment of the source unit of the oil. As such,  $\text{Pr}/\text{Ph}$  values of  $<1.0$  and  $>3.0$  are suggested to indicate either an anoxic or oxic depositional environment, respectively (Didyk et al., 1978; Hunt, 1995; Peters et al., 2005). The  $\text{Pr}/\text{Ph}$  ratios of the Halahatang oils (0.68 - 0.97) (Fig. 5B) suggest that the oils are derived from a source unit deposited under predominantly anoxic conditions in a marine environment. The similar ratios of tricyclic terpanes (e.g.,  $\text{C}_{23}/\text{C}_{21}$  tricyclic terpanes ( $\text{C}_{23}\text{TT}/\text{C}_{21}\text{TT}$ ) ( $\sim 2.05$ ),  $\text{C}_{23}/\text{C}_{24}$  tricyclic terpanes ( $\text{C}_{23}\text{TT}/\text{C}_{24}\text{TT}$ ) ( $\sim 1.73$ ),  $\text{C}_{19}/\text{C}_{23}$  tricyclic terpanes ( $\text{C}_{19}\text{TT}/\text{C}_{23}\text{TT}$ ) ( $\sim 0.16$ ) and the  $\text{C}_{24}$  tetracyclic /  $\text{C}_{26}$  tricyclics terpane ( $\text{C}_{24}\text{TET}/\text{C}_{26}\text{TT}$ ) ( $\sim 0.45$ ) (Table 2) (Fig. 5C)) suggest that the oils belong to one family. The low ratios of  $\text{C}_{19}/\text{C}_{23}$  tricyclic terpane ( $\text{C}_{19}\text{TT}/\text{C}_{23}\text{TT}$ ) (0.12 - 0.20), and  $\text{C}_{24}$  tetracyclic /  $\text{C}_{26}$  tricyclics terpane ( $\text{C}_{24}\text{TET}/\text{C}_{26}\text{TT}$ ) (0.42 - 0.49) suggest that the oil samples are derived from a source containing marine derived organic matter (Bao et al., 2012; Peters and Moldowan, 1993a; Zumberge, 1987) (Table 2)(Fig. 5C). In addition, the similar  $\text{C}_{21}/\text{C}_{22}$  sterane ( $\text{C}_{21}\text{P}/\text{C}_{22}\text{HP}$ ) ratio ( $>3.0$ ) of the oil samples is also indicative that the oils belong to the same family (Table 2). The relative abundance of the  $\text{C}_{27}$ ,  $\text{C}_{28}$ ,  $\text{C}_{29}$  regular steranes are used to constrain the source types, with the  $\text{C}_{27}$  sterane being linked to a marine planktonic source and the  $\text{C}_{29}$  sterane, although they can be derived from algae, is mainly sourced from higher terrestrial plants (Peters and Moldowan, 1993a). The V-shape distribution of the  $\text{C}_{27}$ ,  $\text{C}_{28}$ ,  $\text{C}_{29}$  regular steranes, with  $\text{C}_{27}$  possessing the largest component on the Halahatang oils

379 imply that the organic matter of the source has an alga source (Fig. 4) (Peters and  
380 Moldowan, 1993a). As Ts and diahopane are more resistant to thermal stress than Tm and  
381 hopane, the  $Ts / (Ts+Tm)$  and diahopane/ hopane ( $C_{30}DH/C_{30}H$ ) ratios can provide  
382 insights to the level of oil maturity, with lower ratios equating to lower levels of maturity  
383 (Lu et al., 2010; Peters and Moldowan, 1993a). The  $Ts / (Ts + Tm)$  (~0.34) and  
384  $C_{30}DH/C_{30}H$  (~0.09) ratios of the Halahatang oil samples indicate the oils are low to  
385 moderate maturity (Table 2). The  $C_{29}$  sterane is also a vital biomarker to determine the  
386 level of hydrocarbon maturity (Brooks and Welte, 1984; Peters and Moldowan, 1993a).  
387 The value of  $C_{29}\alpha\alpha/20S/(20S+20R)$  (~0.43) and  $C_{29}\beta\beta/(\beta\beta+\alpha\alpha)$  (~0.57) of the Halahatang  
388 oils (Fig. 5E) equates to a vitrinite reflectance ( $R_o$ ) values of ~0.8 and 0.9, and implies  
389 the oils are within the oil window maturity. The unresolved complex mixture (UCM) of  
390 the gas chromatograms shown by samples Ha9, Ha701 and XK4-3, as well as, the  
391 presence of  $C_{29}$  25-nor-hopane, with  $Nor25H/C_{30}H$  ratios of ~0.98, indicate the  
392 Halahatang oils have suffered biodegradation (Table 2) (Fig. 5F) (Wenger and Isaksen,  
393 2002). However, the relatively complete *n*-alkane compositions of the unresolved  
394 complex mixture indicate a second oil migration and accumulation event may have  
395 occurred in the Halahatang oilfield (Lu et al., 2008; Xiao et al., 2013; Zhu et al., 2012)  
396 (Fig. 4). In the case of the Ha15-2 oil, although the majority of biomarker parameters  
397 possess similar characteristics to the other four Halahatang oil samples, its distinct  
398 maturity ( $Ts / (Ts+Tm) = 0.03$ ) and biodegradation related parameters ( $C_{29}Nor_{25}H/C_{30}H =$   
399 ~0.15) (Table 2) indicate that sample Ha15-2 may have experienced alteration after the  
400 oil reservoir formed. However, as there is no obvious UCM found and that the sample  
401 possesses the lowest  $C_{29}Nor_{25}H/C_{30}H$  value (0.15) (Fig. 4), thermal degradation rather

than the biodegradation may be the main alteration mechanism. In summary, the GC-MS data show that the Halahatang oils belong to one family, derived from the same source that was deposited in a marine environment. In general, the oils are of low to moderate maturity, biodegraded, and may have experienced second hydrocarbon migration event. The organic geochemical differences of oil sample Ha15-2 indicate that some of the oil within the Halahatang oilfield may have undergone alteration after oil generation.

## **5.2 Petroleum evolution timing constraints of the Halahatang Oilfield**

Fluid inclusions represent micron scale samples of the fluids (oil, gas and water) that migrated through and interacted with the host rocks during the evolution of a hydrocarbon system in sedimentary basins (Cooley et al., 2011). Fluid inclusion studies can play a key role in developing pressure, temperature, volume and composition models of fluid (oil and aqueous fluids) dynamics in petroliferous basins. Furthermore, fluid inclusion studies are critical to the understanding of petroleum migration and accumulation, and can help to predict the distribution of petroleum resources (Aplin et al., 1999; Bodnar, 1990; Bourdet et al., 2010; Oxtoby et al., 1995; Pironon, 2004; Teinturier et al., 2002). This study uses aqueous, and hydrocarbon bearing fluid inclusions to help elucidate the history of petroleum and aqueous fluid dynamics in the Tarim basin.

The majority of the hydrocarbon-bearing fluid inclusions of this study exhibit yellow fluorescence, however, some green, and blue fluorescing inclusions are also encountered (Fig. 3). This indicates that the majority oil in the fluid inclusions is of low maturity. Indeed, hydrocarbons of different maturity or having experienced multiple migration events commonly exhibit a range of fluorescence colors (Burruss, 1985; Chen, 2014;

McLimans, 1987; Stasiuk and Snowdon, 1997). Therefore, unlike the previous fluid inclusion results from the Yingmaili oilfield, west of the Halahatang oilfield (Zhu et al., 2013a), the fluid inclusions of this study indicate that the Halahatang oilfield experienced a complex history of hydrocarbon evolution. For example, the blue fluorescing high maturity oil in some of the hydrocarbon bearing inclusions, although uncommon, maybe related to late stage hydrocarbon migration (Guo et al., 2016; Shi et al., 2015; Su et al., 1991).

The Th-salinity bivariate plot (Fig. 6B) for the aqueous FIs defines two groups: a low Th (<100 °C) with <15 wt % NaCl eq. fluid and a fluid with Th >110 °C and a salinity range between ~6 and 22 wt % NaCl eq. This division may reflect at least two stages of aqueous fluid movement. The broad range in Th values of the hydrocarbon bearing inclusions (~24 - 122 °C) may reflect post-entrapment changes to the inclusion bearing fluid. This is further supported by the positive correlation between the vapor bubble volume percent (Fv % at 20 °C) and the Th values (Fig. 6C) (Bourdet et al., 2008). The measured inclusions with the highest Th (>100 °C) also have the highest Fv (13.9 %) values indicating post-entrapment modification. Furthermore, homogenization temperatures can decrease due to post-entrapment thermal cracking (Okubo, 2005). In our study, the two hydrocarbon bearing inclusions with the lowest Th values (~25 °C and 43 °C) also possess the highest CH<sub>4</sub> volume (~95 and 84 μm<sup>3</sup>, Table 3), which may indicate that some of the oils in the Halahatang oilfield have experienced thermal cracking. In general, the Th of hydrocarbon bearing fluid inclusions can also be modified by other post-entrapment events or processes e.g.: necking-down and re-equilibration (Bourdet et al., 2008; Larson et al., 1973). Therefore, the Th of the aqueous fluid

inclusions that are coeval with the hydrocarbon-bearing inclusions are used to constrain the trapping temperature of the fluid that is saturated with CH<sub>4</sub> (Nedkvitne et al., 1993; Visser, 1982). The isochores and isopleths for the aqueous inclusions are plotted as the dash-point lines in P-T space (Fig. 7C). For comparison, isochores are also drawn for non-coeval aqueous inclusions in Figure 7C. The aqueous inclusions coeval with the hydrocarbon-bearing inclusions display a unimodal Th distribution (~61 to 102 °C; mean Th = 82.1 °C, n = 23) (Fig. 7B). Two Th values were used to model the fluid trapping conditions (Fig. 7C) i.e. Th of 83.2 °C (a coeval aqueous inclusion) the other represents the mean Th value (~82.0°C). The solid lines in Figure 6C are the isochores (and isopleths) for the three oil bearing inclusions plotted in Figure 5C. The intersections of the isochores for the oil-bearing inclusion (solid lines, Fig. 7C) and the selected coeval aqueous inclusions provide estimated fluid trapping temperatures of 100 to 110 °C and fluid trapping pressures of ~39 to 59 MPa (Fig. 7C).

Basin modelling coupled with fluid inclusion analysis has been widely applied to constrain the timing of hydrocarbon charging in petroleum systems (Cao et al., 2006; Guo et al., 2012; Roberts et al., 2004). In the Halahatang depression, basin modelling based on wells Ha601 and Ha9 suggest that the Early Palaeozoic source units were buried to ~3500 m (11,483 ft) and underwent hydrocarbon maturation between the Carboniferous and Permian (Zhang et al., 2007a; Zhu et al., 2012). Plotting the modelled Th data from this study with the previous basin model, one prolonged oil migration event during the Permian is proposed (Fig. 7A), which agrees with previous fluid inclusion studies of the Halahatang depression that also indicated the migration of hydrocarbons during the Late Permian (Xiao et al., 2012).

Authigenic illite is one of the last phases formed mineral cements prior to hydrocarbon migration into a sandstone reservoir (Hamilton et al., 1989). If the displacement of an aqueous pore fluid is replaced by hydrocarbons this leads to the cessation of illite formation (Lee et al., 1985). The last formed illite can be used to determine the maximum timing of hydrocarbon emplacement or migration (Hogg et al., 1993). Among the many diagenetic clay mineral products amenable for geochronology, illite is the only commonly occurring diagenetic mineral in sandstone reservoirs that contain sufficient long-lived radioisotope ( $^{40}\text{K}$ ), which permit the determination of its formation age (Hamilton et al., 1989). Although there is no sandstone reservoir in the Halahatang depression, in the Yingmaili Oilfield, ~30 km (~18.6 mi) northwest of the Halahatang depression, the Silurian Keping Formation sandstone reservoir is well-developed (Li et al., 2009). This sandstone strata is a reservoir to bitumen/oil and gas (Zhang and Luo, 2011). Hydrocarbons of the Silurian Keping Formation share a similar origin to hydrocarbons reservoir in Ordovician strata and thus may have charged at the same time (Zhu et al., 2013a). Published illite K-Ar isotope data in the Keping Formation sandstone reservoir (Zhang and Luo, 2011; Zhu et al., 2012) could aid in understanding the petroleum evolution (migration) in the Halahatang oilfield. Seven sandstone samples from different wells (YM11, YM34, YM35, YM35-1) and depths (Fig. 1C) show a decreasing age trend from the northwest ( $293 \pm 2$ ) to the southeast ( $255 \pm 3$  Ma) (Zhang and Luo, 2011; Zhu et al., 2012). The much younger illite K-Ar date than the deposition age of the Keping Formation indicates the sandstone samples contain no or little detrital illitic contamination and that the K-Ar date obtained from the fine fraction should approximately reflect the timing of diagenetic illite formation. Except for the Yingmaili

oilfield which is near to the Halahatang oilfield, other K-Ar dates in Tarim Basin, e.g., the Hadexun oilfield in the Northern Depression and Tazhong oilfield, respectively ~50 km (~31 mi) and ~100 km (~62 mi) south to the Hahalatang depression (Fig. 1B), also possess illite K-Ar dates of ~250 Ma and ~230 Ma, respectively (Zhang et al., 2007b; Zhu et al., 2013b). All the illite K-Ar dates (~280 - 230 Ma) from the central and northern Tarim Basin coincide with the above fluid inclusion and basin modelling data that show the major hydrocarbon migration and accumulation occurred predominantly during between the Mid to Late Permian.

Petroleum evolution is a complex process including oil generation, migration and finally accumulation or destruction. Oil generation, which leads the whole evolution process, is one key factor. Previous research has shown that oil generation is a multi-step procedure involving bitumen formation from the kerogen and oil generation from the bitumen, and that these two steps are closely related (Lewan, 1985). Oil/bitumen/pyrobitumen Re-Os analysis, which is a new and challenging method, has shown potential in constraining the absolute timing of the oil/bitumen/pyrobitumen generation (Cumming et al., 2014; Finlay et al., 2011; Ge et al., 2016; Lillis and Selby, 2013; Liu et al., 2018; Selby and Creaser, 2005). Although the GC-MS analysis and fluid inclusion analysis indicate that some oil samples in this study have suffered from biodegradation and secondary migration, however, previous research has shown that biodegradation do not significantly affect the Re-Os systematics of oil (Lillis and Selby, 2013). Moreover, and if the oils generated during one period, then multiple oil migration episodes following the generation will not disturb the hydrocarbon Re-Os system (Finlay et al., 2011; Lillis and Selby, 2013; Selby et al., 2005).



The Re-Os data for all oil samples, except sample Ha15-2, yield a Model 3 Re-Os age of  $285 \pm 48$  Ma ( $Os_i = 1.08 \pm 0.20$  [18.5 %], MSWD = 6.1). The large age uncertainty (~17 %) and MSWD value is beyond that associated with analytical uncertainty (~1.0), and is considered to be directly related to the variation in the initial  $^{187}\text{Os}/^{188}\text{Os}$  ratio ( $Os_i$ ) of the sample set (Cohen et al., 1999; Ludwig, 2008) (Fig. 8; Table 4). Given that oils are generated from a source horizon that can be both stratigraphically (10s to 100s of m/ft) and geographically (10s to 100s kilometers/miles) expansive, the ability for oil sampled across a reservoir to possess the same initial  $Os_i$  ratio can be challenged (see (Lillis and Selby, 2013)), but can also exhibit a limited range in values ((Liu et al., 2018 and references therein). The variation in  $Os_i$  values could also relate to the oil sampled being associated with difference stages of the oil generation of a petroleum system (Liu et al., 2018).

The ~285 Ma Re-Os date, including its uncertainty of 48 Ma, is in good agreement with the understanding of the timing of oil generation in northern Tarim Basin (Zhu et al., 2013a; Zhu et al., 2012). Basin modelling both in the Halahatang and Yingmaili oilfields of the northern Tarim Basin, suggest that a Paleozoic source was buried to ~3000 m (~9842 ft) and underwent hydrocarbon maturation during the Late Carboniferous to Early Permian (Zhu et al., 2013a; Zhu et al., 2012). Although possessing a relatively large uncertainty (48 Ma), the ~285 Ma Re-Os date, which is nominally slightly older than the oil migration / accumulation timing constrained by basin modelling coupled with fluid inclusion analysis and the reservoir illite K-Ar dates (see above), suggests that the Re-Os date represents the best absolute estimate for the timing of oil generation in the Halahatang oilfield.

The oil sample Ha15-2 plots to right of the defined best-fit line (isochron) of the bulk of the Re-Os data for the oil sample set (Fig. 8). In comparison to the other oil samples, sample Ha15-2 also possesses different parameters in biomarker analysis ( $C_{29}NOR_{25}H/C_{30}H = 0.15$ ,  $Ts/(Ts+Tm) = 0.03$ ) indicating the sample has experience post-generation alteration. Furthermore, although of limited abundance, the presence of oil-bearing fluid inclusions with high Th ( $>120^{\circ}C$ ) and a  $CH_4$  volume of  $\sim 90 \mu m^3$  of this study (Table 3) also indicate thermal cracking may have occurred in regions in the Halahatang oilfield. Moreover, the basin modelling shows that the Paleozoic strata in this area have been buried to  $\sim 7000$  m ( $\sim 22,966$  ft) since the Late Neogene (Fig. 7A) (Zhu et al., 2012), and that during the last  $\sim 10$  Myrs the high temperatures ( $>150^{\circ}C$ ) may have led to thermal cracking of oil, particular in deeper parts, of the Halahatang oilfield (eg, Ha 15-2 oil). Previous work on TSR affected oil from the Manderson, South of the Bighorn Basin (Lillis and Selby, 2013) and pyrobitumen from Majiang - Wanshan reservoir, South China (Ge et al., 2016) have shown that the high temperature controlled thermal cracking can reset the Re-Os systematics in the hydrocarbons (oil/bitumen). The high Th and  $CH_4$  volume evidence from fluid inclusions, and basin modelling suggest that the thermal cracking which could lead to gas formation (Hill et al., 2003; Huc et al., 2000) may have resulted in the Re-Os characteristics shown by oil sample Ha15-2. Additional research on similar oils to Ha15-2 from the Halahatang oilfield will be necessary to see if the Re-Os systematics are still being effected by thermal cracking as it has been suggested the closure temperature of Re-Os in thermal cracked oil is  $\leq 120^{\circ}C$  (Ge et al., 2016; Lillis and Selby, 2013).

### 5.3 Petroleum evolution in the Halahatang depression

Based on the general tectonic evolution of the Tarim Basin, and combining the oil Re-Os dating and fluid inclusion analysis of this study from the Halahatang depression, and previous basin modeling and illite K-Ar isotope dates from Tarim Basin, the petroleum evolution in the Halahatang depression can be summarized as follows. Exhumation of Silurian strata driven by the Caledonian Orogeny during the Devonian (Fig. 9A) (Lin et al., 2015), the Tarim Basin transferred into an extensional environment between the Carboniferous and Permian (Zhang et al., 2007a). The continued subsidence during this period led to the burial (>3500 m) (>11483 ft) of the Paleozoic source units (Cambrian to Ordovician shales/mudstone) and oil generation during the Early Permian (Fig. 9B) (~285 Ma Re-Os oil date). The closure of the Tian Shan Sea (Late Hercynian tectonic event) caused a phase of uplift and exhumation during the Mid-Late Permian that resulted in the cessation the oil generation (Lin et al., 2015; Zhang et al., 2007a). However, tectonic instability, as well as, the simultaneous formed faults provided pathways for oil migration and accumulation (Zhu et al., 2013c). Both basin modelling and coupled fluid inclusion analysis of this study and previous illite K-Ar dating (~280 - 230 Ma) within central and northern Tarim Basin (Zhang et al., 2007b; Zhang and Luo, 2011; Zhu et al., 2012; Zhu et al., 2013b) show that hydrocarbon migration and accumulation occurred predominantly during the Mid-Permian, but also during the Early Triassic (Fig. 9C). The Tarim Basin changed to a continental sedimentary depositional environment as of the Mesozoic (Zhang and Huang, 2005). The basin burial history of the Halahatang depression shows a continuous sedimentary deposition since the Late Triassic including a rapid sedimentation since the Neogene (Fig. 9D). This has resulted in the deep burial (~7000 m) (22965 ft) of the Ordovician reservoirs. The deep burial and high temperatures

( $>150\text{ }^{\circ}\text{C}$ ) are suitable for oil cracking to occur. Both the high Th and  $\text{CH}_4$  volume observed in oil-bearing fluid inclusion and the oil Re-Os isotope characteristics of Ha 15-2 oil show evidence of thermal cracking in parts of the Halahatang oilfield. However, the organic geochemistry (Fig. 4) (Table 2), fluid inclusion analysis (Fig. 3) (Table 3) and Re-Os data (Fig. 8) of this study suggests that thermal cracking of oil in the Halahatang depression is neither prolonged nor widespread.

## 6 Conclusions

Combining the organic geochemical analysis, fluid inclusion analysis and Re-Os isotope analysis of the Halahatang oilfield, in addition to previously published basin modeling and illite K-Ar dating in Tarim Basin, the petroleum evolution of the Halahatang oilfield, Northern Tarim Basin is quantitatively constrained. The organic geochemistry analyses show the oil samples belong to same family, with the oils derived from a source deposited in an anoxic marine environment, possess low to middle maturity and have undergone limited biodegradation. The  $\sim 285\text{ Ma}$  Re-Os date coincides with the hydrocarbon trap evolution, the Cambrian-Ordovician source rock maturation history (Zhang et al., 2007a), basin modelling result for the Halahatang depression, and the nearby Yingmai Basin indicating that the Re-Os data records the timing of oil generation. Traditional basin modelling coupled with fluid inclusion analysis are in good agreement with published illite K-Ar dates ( $280 - 230\text{ Ma}$ ) (Zhang et al., 2007b; Zhang and Luo, 2011; Zhu et al., 2012; Zhu et al., 2013b) in the Tarim basin and suggest that oil migration/accumulation occurred during the Mid-Permian to Early Triassic. The Re-Os geochemistry of Ha15-2, and oil-bearing fluid inclusion analysis suggest that some oils in

the Halahatang depression have experienced thermal cracking since the Neogene. Integrating fluid inclusion analysis, reservoir illite K-Ar dating and Re-Os oil analysis, this work quantitatively establishes the entire petroleum evolution (generation, migration/accumulation) process in the Halahatang depression of the northern Tarim Basin. In addition to Tarim basin, the coupled analysis of Re-Os oil geochronology with K-Ar dating and fluid inclusions is also applicable to petroleum systems worldwide to aid in the understanding of both the temporal and spatial evolution of hydrocarbon systems.

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#### **Figure captions**

Fig. 1. (A) Location of the Tarim Basin, China. (B) Structural unit distribution of the Tarim Basin. (C) Regional map of the Halahatang depression, Yingmaili Uplift, Luntai Uplift, Lunnan Uplift and the Northern Depression (Substantially modified from (Zhu et al., 2012)). Also shown are the sample locations for the Re-Os analysis of this study and the illite K-Ar analysis (Zhang and Luo, 2011; Zhu et al., 2012). The dash line A-A' shows the location of the cross section profile presented in Figure 8.

Fig. 2. Combined Stratigraphic sequences, hydrocarbon system and tectonic events in the north Tarim Basin. Substantially modified after Chang et al., 2013a; Lin et al., 2015 and Zhang and Huang, 2005

Fig. 3. Photomicrographs of (A) an example of a liquid-rich, two-phase (L+V; L>V) aqueous inclusion (~10 microns in longest dimension) in sample Ha9 and (B) an example of a two-phase hydrocarbon bearing fluid inclusion (~10 microns in longest dimension) in sample Ha9. (C, D) examples of hydrocarbon bearing fluid inclusions under

1001 fluorescent light in sample Ha9.

1002

1003 Fig. 4. GC,  $m/z$  191 and  $m/z$  217 mass chromatogram of the oil samples (Ha9, Ha11,  
1004 Ha701, XK4-3 and Ha15-2) in Halahatang oilfield, northern Tarim Basin.

1005

1006 Fig. 5. (A). plots of Pristane/ $nC_{17}$  and Phytane/ $nC_{18}$  alkane ratios for sampled oils. (B)  
1007 Histogram of the Pr/Ph ratios for sampled oils. (C) Distribution of the tricyclic terpanes  
1008 (ratios of  $C_{23}TT/C_{21}TT$ ,  $C_{23}TT/C_{24}TT$ ,  $C_{19}TT/C_{23}TT$  and  $C_{24}TET/C_{26}TT$ ) for the samples  
1009 oils. (D) Histogram of the Ts/Ts+Tm ratio for the sampled oils. (E) Plots of  
1010  $C_{29}aaa_{20S}/(20S+20R)(\sim 0.43)$  and  $C_{29}\beta\beta/(\beta\beta+aa)(\sim 0.57)$  of the sampled oils. (F)  
1011 Histogram of the  $C_{29}Nor_{25}H/C_{30}H$  ratio for the sampled oils.

1012

1013 Fig. 6. (A) A Th frequency distribution histogram of aqueous, coeval (with hydrocarbon-  
1014 bearing fluid inclusions) aqueous and hydrocarbon-bearing fluid inclusions in sample  
1015 Ha9. (B) A Th-salinity bivariate plot of aqueous fluid inclusions in sample Ha9. (C) A  
1016 bivariate plot of Fv (at 20°C) Th for three selected hydrocarbon bearing fluid inclusions  
1017 from sample Ha9. Seven oil types after (Bourdet et al., 2008) are also plotted for  
1018 reference indicating that the fluid inclusion hosted oils in sample Ha9 similar in  
1019 composition to N. America volatile oils (NA = North America).

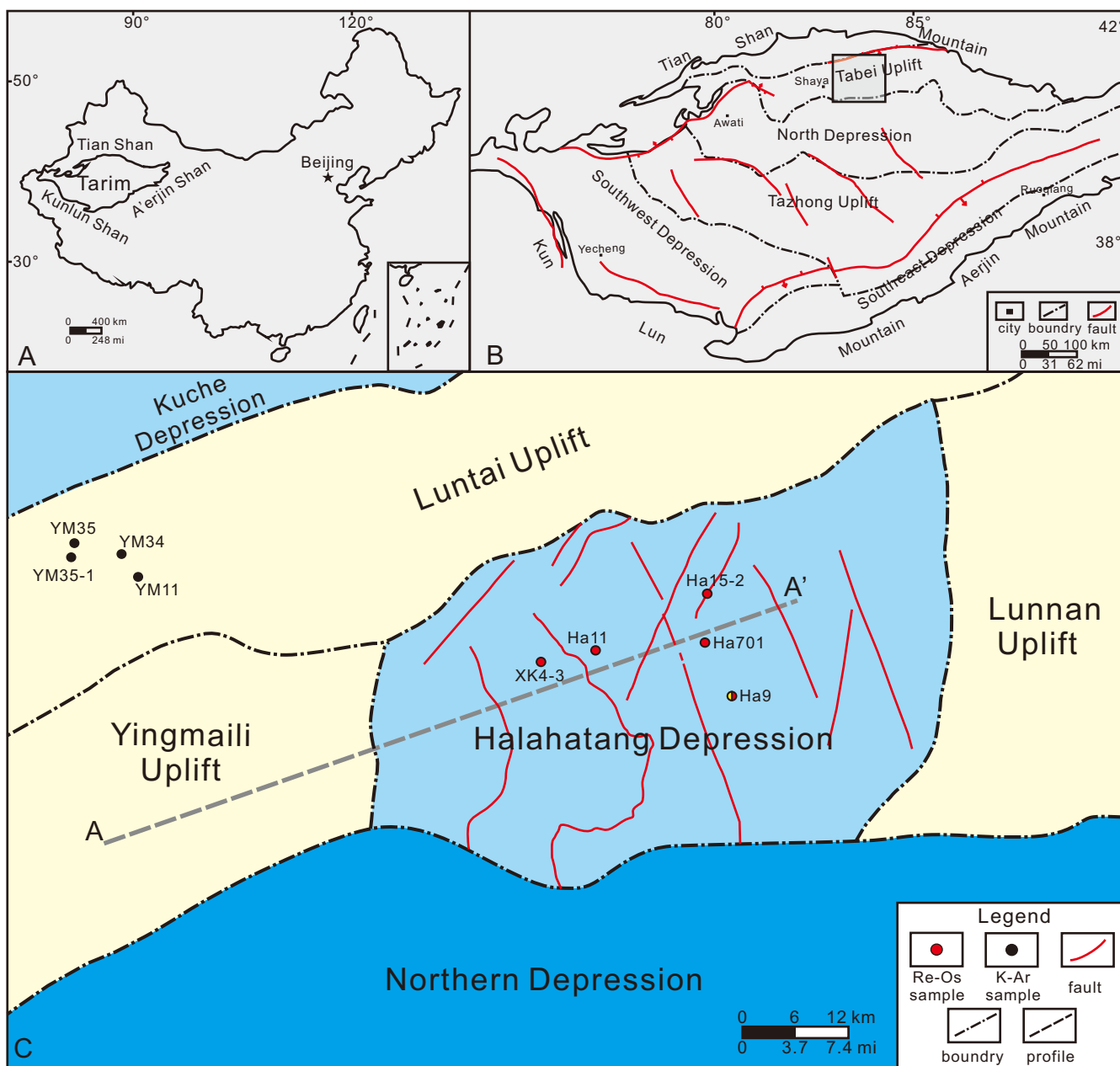
1020

1021 Fig. 7. (A) A burial history temperature-time plot for the Halahatang oilfield, showing the  
1022 key time for oil migration and or accumulation. (B) A Th frequency distribution  
1023 histogram for aqueous fluid inclusions coeval with hydrocarbon-bearing fluid inclusions

in sample Ha 9. (C) A P-T plot of isopleths and isochores for aqueous and hydrocarbon bearing fluid inclusions in Ha9. Isopleths and isochores of the hydrocarbon fluids are determined using PIT modeling (Montel, 1993; Pironon, 2004), whereas isopleths and isochores for the aqueous fluids were generated using (Duan and Mao, 2006). The dash-point lines: coeval aqueous fluid inclusions (PI with AI). The dash lines: non-coeval aqueous fluid inclusions (AI). The solid lines: three hydrocarbon bearing fluid inclusions (PI)(plotted in Fig 5C) and are coeval with the aqueous fluid inclusions. Intersections between the coeval isochores yield estimates of true fluid trapping temperatures and pressures. See text for discussion.

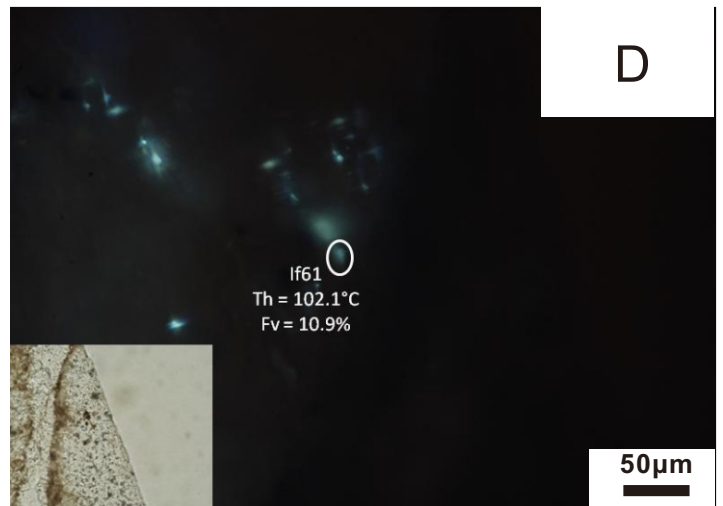
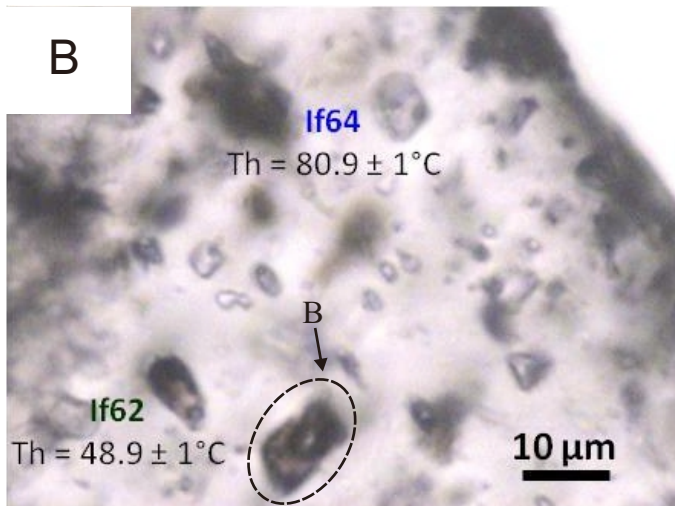
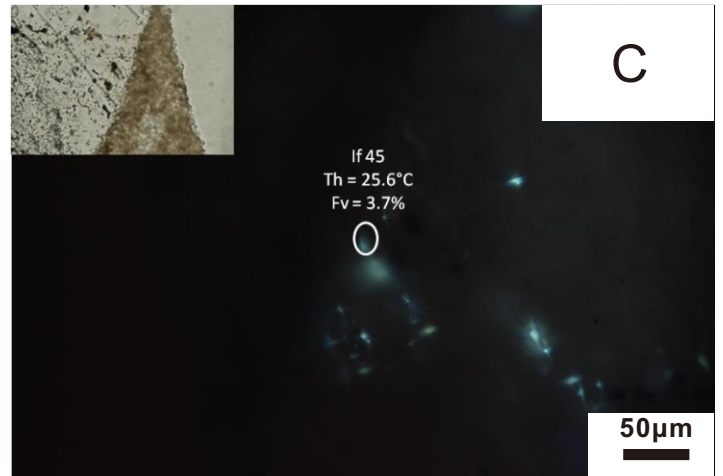
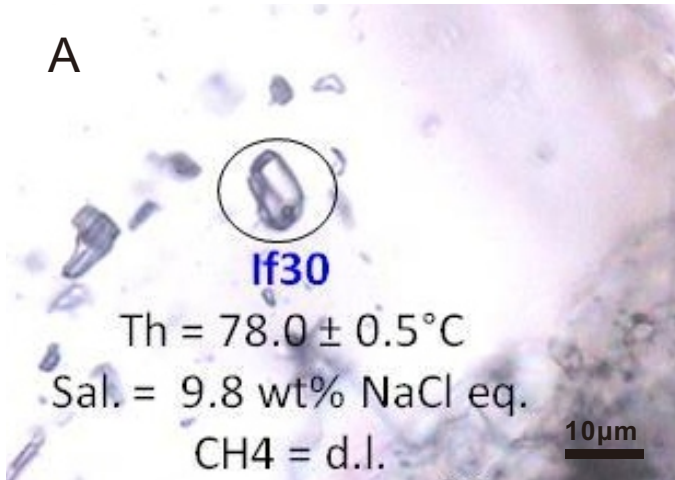
Fig. 8. Re-Os isochron plot of all oil samples Ha9, Ha9rpt, Ha11, Ha701, XK4-3. Data-point ellipses shown at the 2-sigma level absolute uncertainty. The Re-Os data for all samples, except Ha15-2 and Ha15-2rpt, yield a Re-Os date of  $285 \pm 48$  Ma, with an initial  $^{187}\text{Os}/^{188}\text{Os}$  of  $1.08 \pm 0.20$  (MSWB = 6.1). Ha9rpt and Ha15-2rpt are repeated analysis of samples Ha9 and Ha15-2.

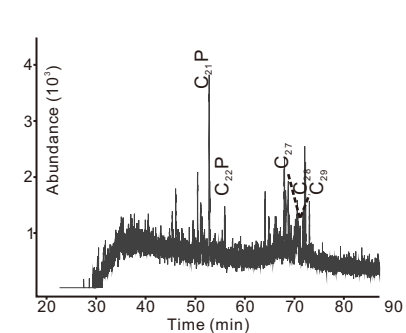
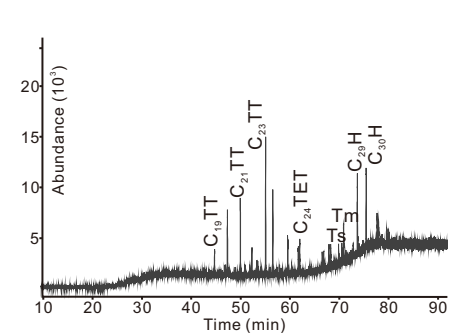
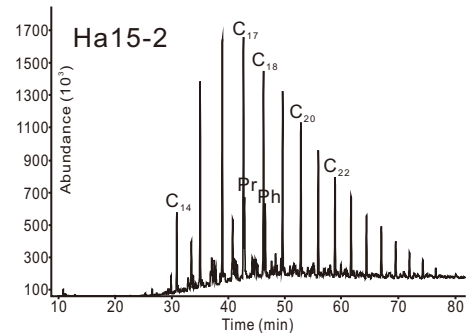
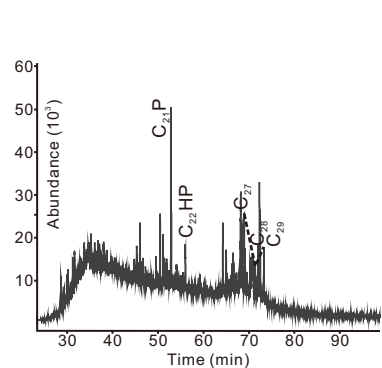
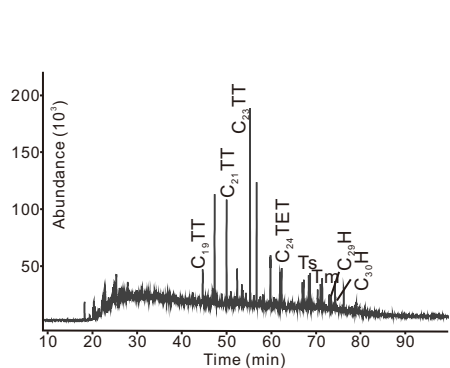
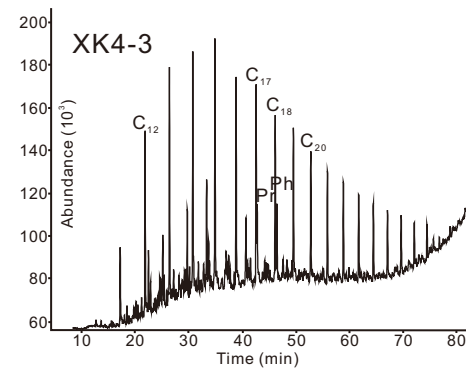
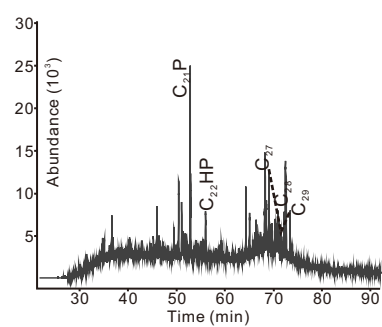
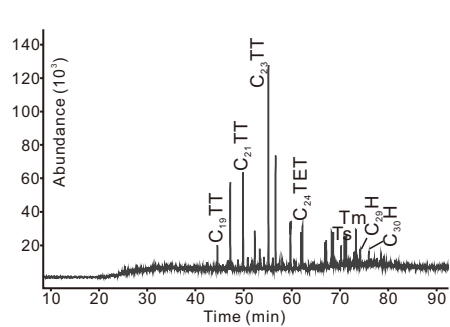
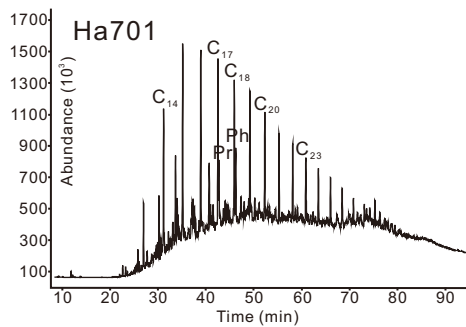
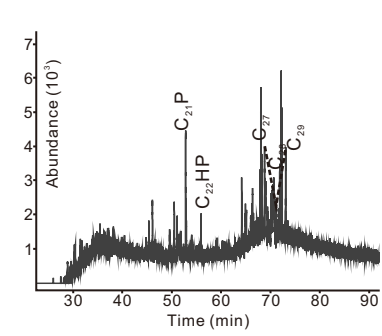
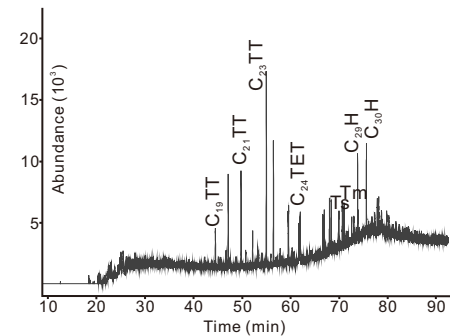
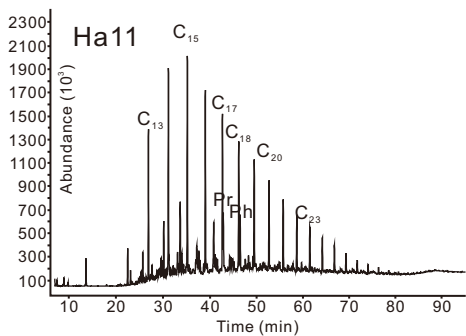
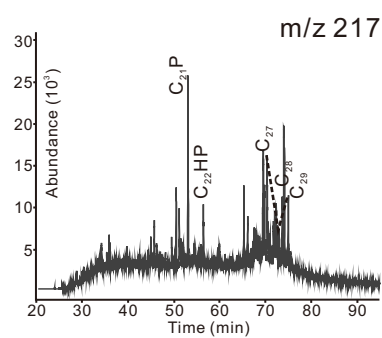
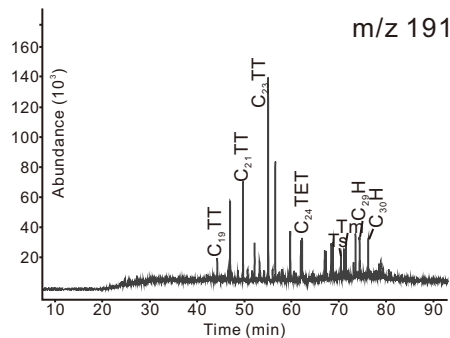
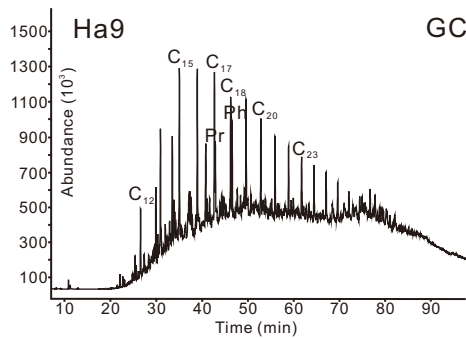
Fig. 9. Petroleum evolution model for the Halahatang oilfield, northern Tarim Basin. (A) Halahatang depression during the Late Silurian. (B). Oil generation during the Early Permian. (C) Oil migration/accumulation process during the Late Permian to Early Triassic. (D) Present day configuration of the Halahatang oilfield after continuous sedimentation since the Late Triassic. See text for discussion.

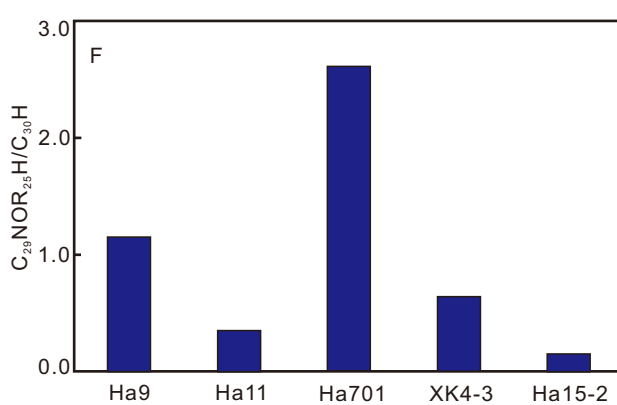
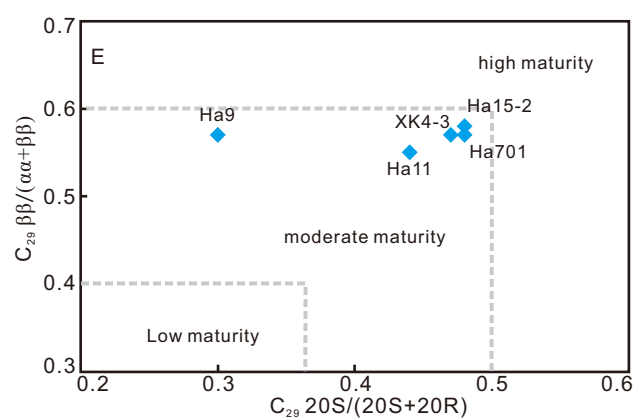
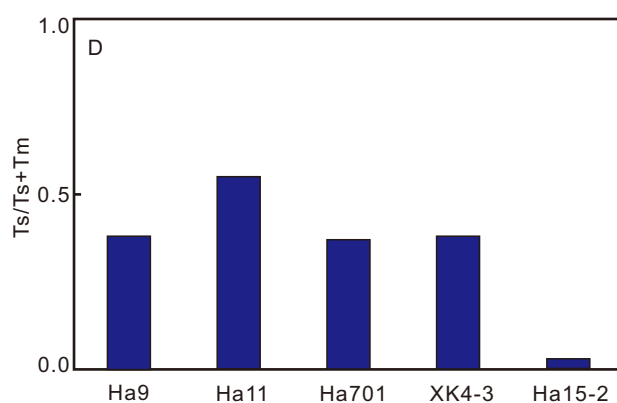
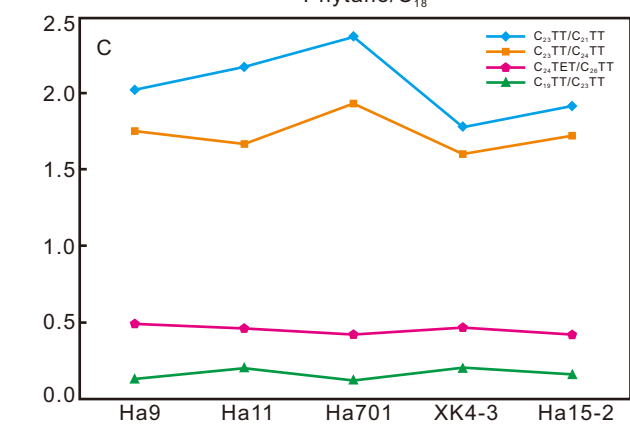
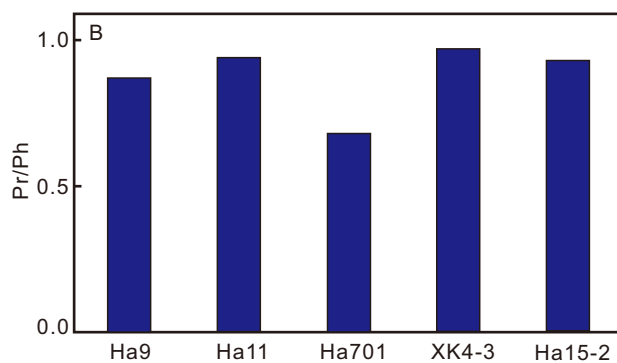
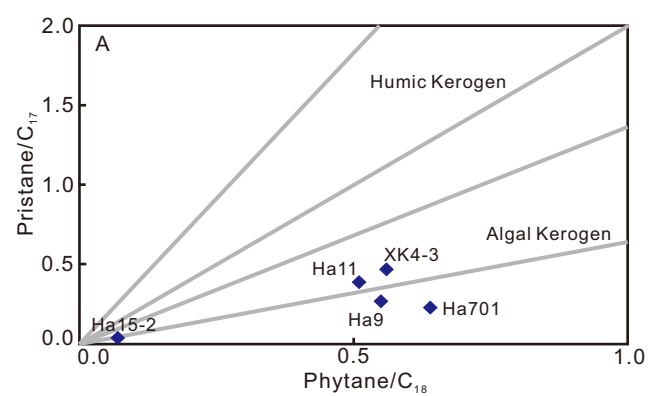


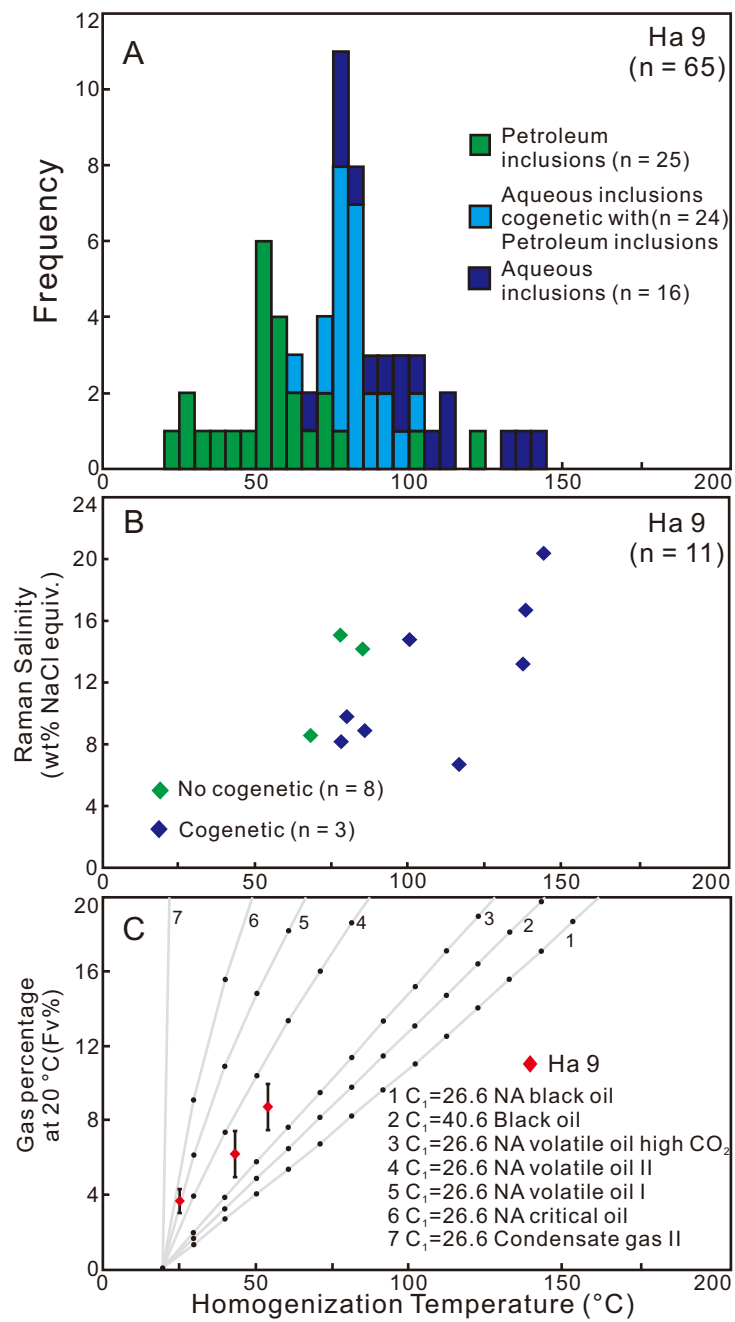


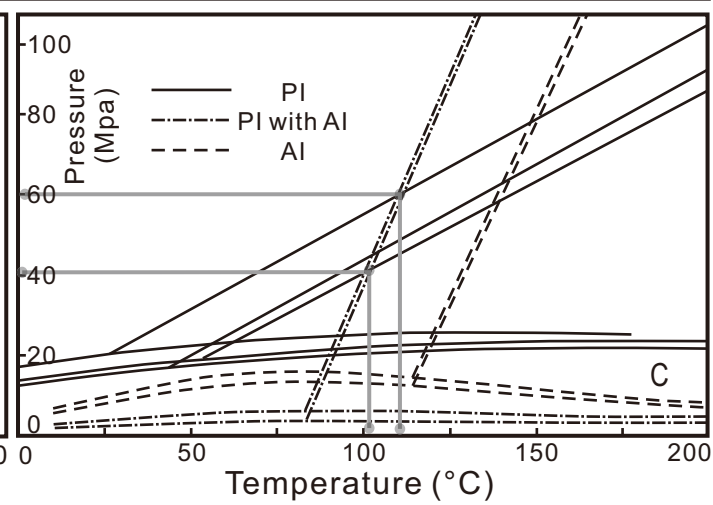
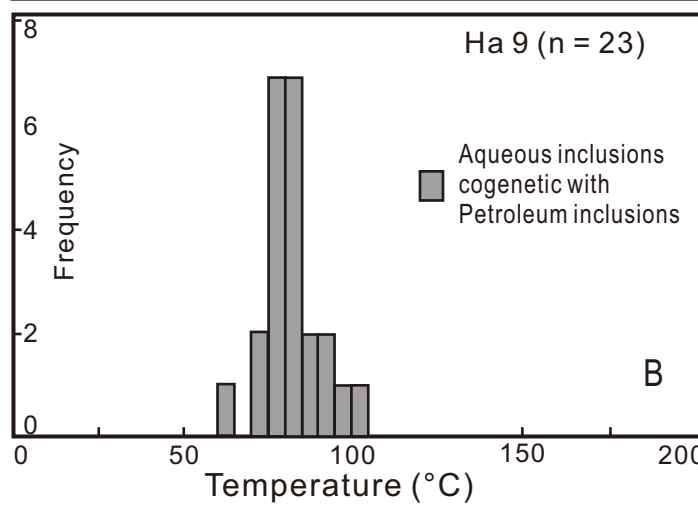
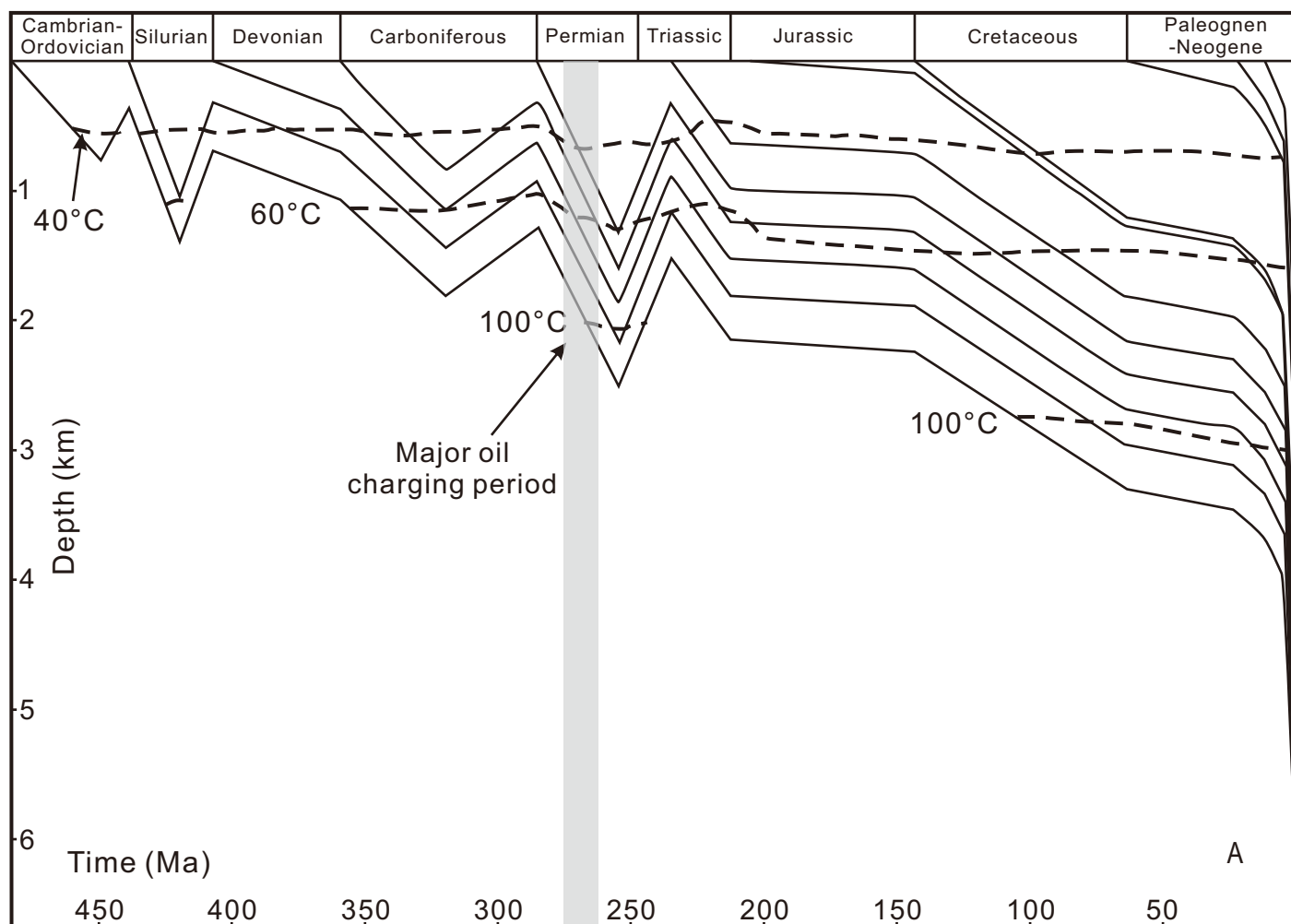
Period	Formation	Lithology	Thickness (m)	Petroleum System	Tectonic Cycle
Quaternary			100-200		
	2.5	Xiyu			
		Kuche			
Neogene		Kangcun	2000 - 4000		
	23	Jidike			
		Suweiya			
Paleogene		Kumugeliemu	100-500		Himalayan Movement
	65				
Cretaceous		Kapushaliang	200-600		
	145				
Jurassic			100-300		Yanshan Movement
	201				
		Halahatang			
Triassic		Akekule	400-600	reservoir	Indosinian Movement
	252	Ketuler			
		Shajingzi			
Permian		Kaipaizileike	300-500		
		Kupukuziman			
	299	Nanmin			
		Xiaohaizi			
Carboniferous		Kalashayi	300-800		
	359	Bachu			
Devonian		Donghetang	0-300	reservoir	Hercynian Movement
	419	Keziertage			
Silurian		Yimugantanwu	0-1000	reservoir	
	444	Tataaiertage			
		Kepingtage			
		Sangtamu		source	
		Lianglitage			
Ordovician		Qiaerbake	500 - 2000		
		Yijianfang		reservoir	
		Yingshan			
	485	Penglaibai			Caledonian Movement
		Qiulitagexia			
		Awatage			
		Shayilike			
Cambrian		Wusonggeer	200 - 1000	reservoir	
		Xiaoerbulake		source	
	542	Yuertusi			
Neo-proterozoic		Qigebulake			
	650	Sugaitebulaike			

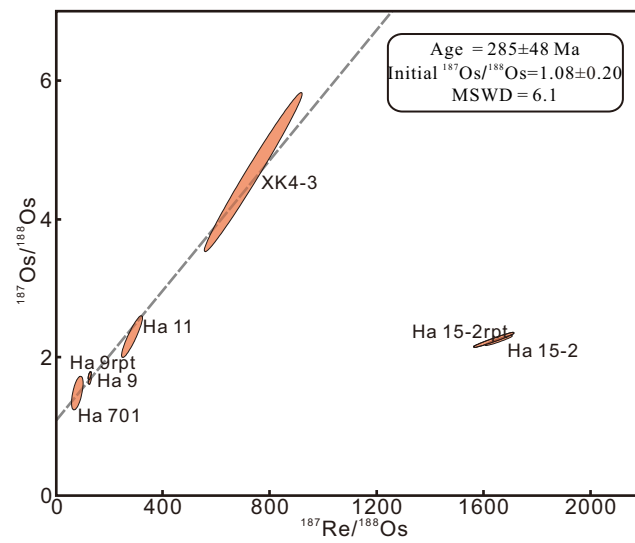












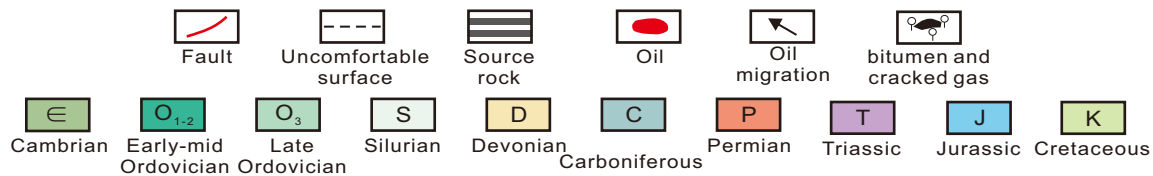
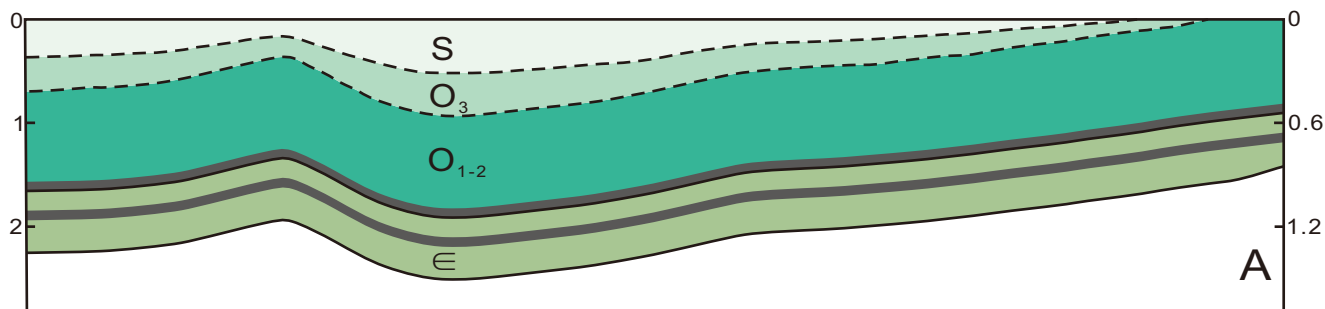
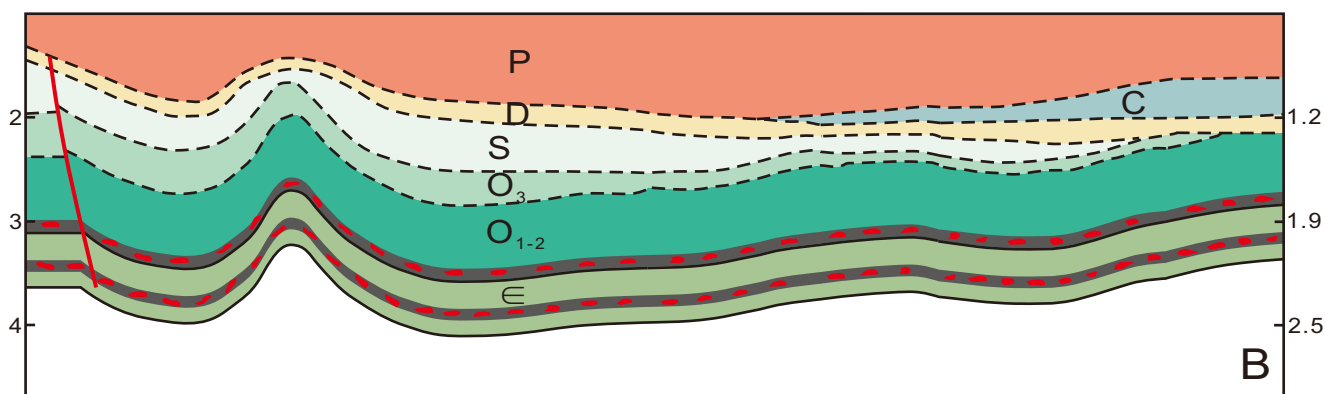
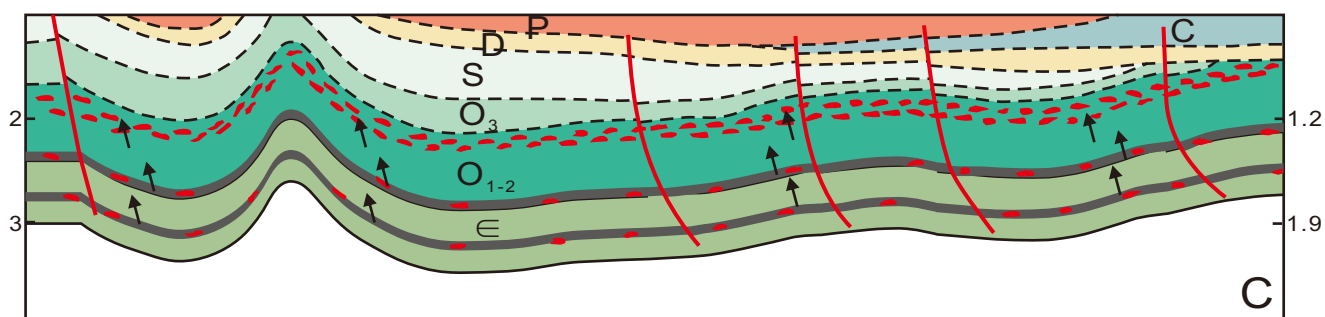
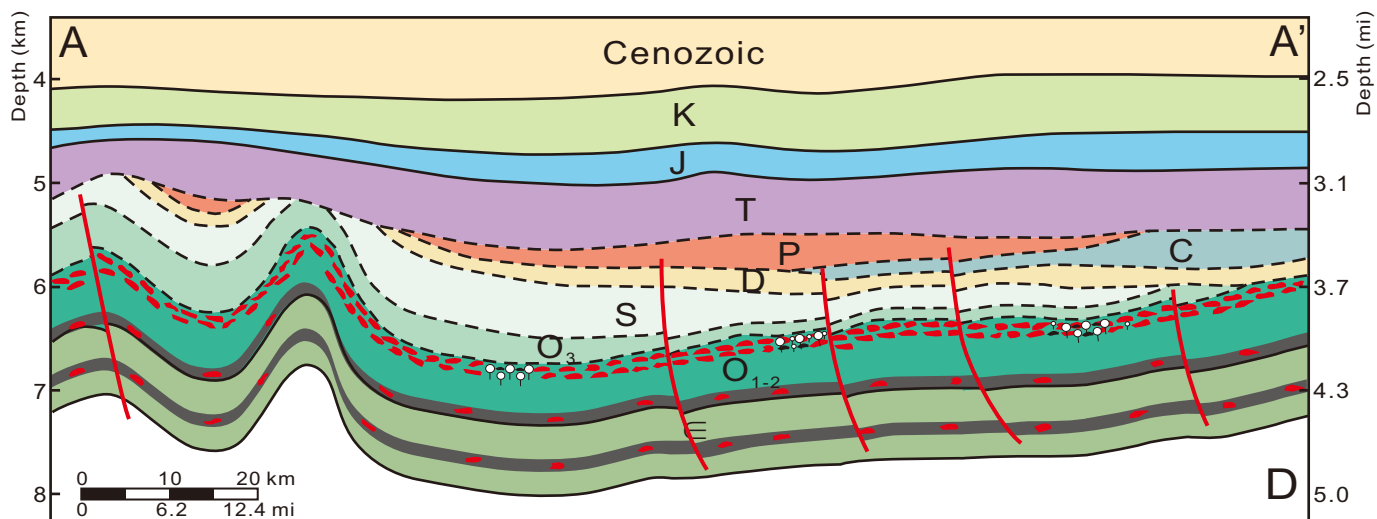




Table 1. Physical properties, sulfur content and component of the oil from the halahatang oilfield.

Well name	Depth (m)	Formation Name	Density (g/cm <sup>3</sup> )		Viscosity (mPa·s, 50 °C)	API vaule	Wax (%)	Sulfur (%)	Saturate (%)	Aromatic (%)	Resin (%)	Asphaltene (%)
			20 °C	50 °C								
<b>Ha 9</b>	6598-6710	Ordovician Yijianfang	0.879	0.859	6.92	29.48	3.80	0.84	57.33	10.00	6.67	23.33
<b>Ha 11</b>	6658-6748	Ordovicia Yijianfang	0.833	0.812	2.62	38.37	6.10	0.50	64.67	14.33	3.67	14.33
<b>Ha 15-2</b>	6559-6598	Ordovician Yijianfang	0.844	0.822	3.15	36.15	8.60	0.63	56.00	18.00	4.33	18.67
<b>Ha 701</b>	6557-6618	Ordovician Yijianfang	1.011	0.992	342.3	8.46	/	0.67	46.92	13.36	3.42	33.56
<b>XK4-3</b>	6834-6850	Ordovician Yijianfang	0.88	0.86	7.96	29.30	5.90	0.59	55.49	26.55	5.24	3.56

/: Not measured

Table 2. The biomarker parameters of the oil samples from Halahatang oilfield.

Sample name	Pr/nC <sub>17</sub>	Ph/nC <sub>18</sub>	Pr/Ph	C <sub>23</sub> TT/C <sub>21</sub> TT	C <sub>23</sub> TT/C <sub>24</sub> TT	C <sub>19</sub> TT/C <sub>23</sub> TT	C <sub>24</sub> TET/C <sub>26</sub> TT	Ts/(Ts+Tm)	GAM/C <sub>30</sub> H
Ha9	0.27	0.55	0.87	2.02	1.75	0.13	0.49	0.38	0.19
Ha11	0.39	0.51	0.94	2.17	1.67	0.20	0.46	0.55	0.12
Ha701	0.23	0.64	0.68	2.37	1.93	0.12	0.42	0.37	0.15
XK4-3	0.47	0.56	0.97	1.78	1.60	0.20	0.47	0.38	0.05
Ha15-2	0.04	0.07	0.93	1.91	1.72	0.16	0.42	0.03	0.07
	C <sub>30</sub> DH/ C <sub>30</sub> H	C <sub>29</sub> NOR <sub>25</sub> H/ C <sub>30</sub> H	C <sub>21</sub> P/C <sub>22</sub> HP	C <sub>27</sub> R (%)	C <sub>28</sub> R (%)	C <sub>29</sub> R (%)	C <sub>29</sub> ααα20S/(20S+20R)	C <sub>29</sub> ββ/(ββ+αα)	
Ha9	0.10	1.15	3.12	56.69	7.56	35.75	0.30	0.57	
Ha11	0.03	0.35	2.98	37.40	20.07	42.53	0.44	0.55	
Ha701	0.13	2.61	4.20	65.37	7.02	27.60	0.48	0.57	
XK4-3	0.10	0.64	5.69	49.64	15.96	34.39	0.47	0.57	
Ha15-2	0.10	0.15	4.15	42.20	22.70	35.11	0.48	0.58	

Table 3. Summary of petrographic observations, microthermometry, Laser Raman Microspectroscopy and CSLM results of aqueous and oil-bearing inclusions for sample Ha9.

Sample Name	Inclusion No.	FI types	types	Th (°C)	Error (°C)	Raman Salinity wt% (NaCl aq.)	Raman Salinity (molal)	CH <sub>4</sub> (molal)	Bulk volume (μm <sup>3</sup> )	Bubble volume (μm <sup>3</sup> )	Fv at 20°C (%)	Relative error %
Ha 9-1A	42	2-phase L-V oil	Calcite cement	52.8	2							
Ha 9-1A	43	2-phase L-V oil	Calcite cement	52.6	1							
Ha 9-1A	44	2-phase L-V aqueous	Calcite cement	98.1	0.2	14.6	3.02	0.1				
Ha 9-1A	45	2-phase L-V oil	Calcite cement	25.6	0.5				94.7	3.5	3.7	17
Ha 9-1B	45b	2-phase L-V oil	Calcite cement	n.m					32.6	1.8	5.1	49
Ha 9-1B	46	2-phase L-V aqueous	Calcite cement	85.8	2							
Ha 9-1B	47	2-phase L-V oil	Calcite cement	25.1	0.3							
Ha 9-1B	48	2-phase L-V aqueous	Calcite cement	71.7	0.3							
Ha 9-1B	49	2-phase L-V oil	Calcite cement	57.2	0.2							
Ha 9-1B	50	2-phase L-V aqueous	Calcite cement	86.7	2							
Ha 9-1B	68	2-phase L-V aqueous	Calcite cement	83.2	0.2	14.2	2.88	0.017-0.026				
Ha 9-1B	69	1-phase L aqueous	Calcite cement			18.6	4.04					
Ha 9-1C	51	2-phase L-V oil	Calcite cement	54.2	0.2							
Ha 9-1C	52	2-phase L-V oil	Calcite cement	24.6	0.3							
Ha 9-1C	53	2-phase L-V oil	Calcite cement	29.2	0.3							
Ha 9-1C	54	2-phase L-V oil	Calcite cement	37	0.2							
Ha 9-1C	55	2-phase L-V aqueous	Calcite cement	83.8	1							
Ha 9-1C	56	2-phase L-V aqueous	Calcite cement	91.6	2							
Ha 9-1C	57	2-phase L-V aqueous	Calcite cement	107.1	2							
Ha 9-1C	58	2-phase L-V aqueous	Calcite cement	77	1							
Ha 9-1C	59	2-phase L-V aqueous	Calcite cement	76.1	1	15.1	3.1	n.m.				
Ha 9-1C	60	2-phase L-V aqueous	Calcite cement	83.3	0.5							
Ha 9-1D	61b	2-phase L-V oil	Calcitized bioclast	122	2							
Ha 9-1D	62	2-phase L-V oil	Calcitized bioclast	48.9	1							
Ha 9-1D	63	2-phase L-V aqueous	Calcitized bioclast	79.5	0.5							
Ha 9-1D	64	2-phase L-V aqueous	Calcitized bioclast	80.9	1							
Ha 9-1D	65	2-phase L-V aqueous	Calcitized bioclast	82.9	1							
Ha 9-1D	67	2-phase L-V aqueous	Calcitized bioclast	79.6	1							
Ha 9-2A	1	2-phase L-V aqueous	Calcitized bioclast	90.6	0.5							
Ha 9-2A	2	2-phase L-V aqueous	Calcite cement	76.1	0.5							
Ha 9-2A	3	2-phase L-V aqueous	Calcite cement	102.3	1							
Ha 9-2A	7	2-phase L-V aqueous	Calcite cement	75.4	1							
Ha 9-2A	8	2-phase L-V aqueous	Calcite cement	74	0.3							
Ha 9-4A	10	2-phase L-	Calcite	77.3	0.3							

		V aqueous		cement					
<b>Ha 9-4A</b>	11	2-phase L-V aqueous	Calcite cement	75.1	1				
<b>Ha 9-4A'</b>	12	2-phase L-V oil	Calcite cement	59.1	0.5				
<b>Ha 9-4A'</b>	13	2-phase L-V oil	Calcite cement	58.1	2				
<b>Ha 9-4B</b>	15	3-phase L-V oil	Calcitized bioclast	50.8	1				
<b>Ha 9-4C</b>	16	2-phase L-V oil	Calcitized bioclast	60	1				
<b>Ha 9-4C</b>	17	2-phase L-V oil	Calcitized bioclast	30.9	1				
<b>Ha 9-4C</b>	18	2-phase L-V oil	Calcitized bioclast	79.9	1				
<b>Ha 9-4D</b>	20	2-phase L-V aqueous	Calcite cement	66.4	0.5	8.6	1.63	n.m.	
<b>Ha 9-4D</b>	21	2-phase L-V aqueous	Calcite cement	66.3	2	9.5	1.82	d.l.	
<b>Ha 9-4D</b>	21b	2-phase L-V aqueous	Calcite cement	117	1	6.7	1.24	0.067-0.075	
<b>Ha 9-4D</b>	21c	2-phase L-V aqueous	Calcite cement	134.3	2	13.2	2.63	n.m.	
<b>Ha 9-4D</b>	22	2-phase L-V oil	Calcite cement	53.5	0.3				29.4 2.6 8.7 14
<b>Ha 9-4E</b>	23	2-phase L-V oil	Calcitized bioclast	68.1	0.5				
<b>Ha 9-4E</b>	24	3-phase L-V oil	Calcitized bioclast	63.4	2				
<b>Ha 9-4E</b>	25	2-phase L-V oil	Calcitized bioclast	43.1	1				84.4 5.6 6.2 20
<b>Ha 9-4E</b>	26	2-phase L-V oil	Calcitized bioclast	51.8	2				
<b>Ha 9-4E</b>	26b	2-phase L-V oil	Calcitized bioclast	n.m.					21.8 1.6 6.7 19
<b>Ha 9-5A</b>	27	2-phase L-V aqueous	Calcitized bioclast	61.2	0.3				
<b>Ha 9-5A</b>	28	2-phase L-V aqueous	Calcitized bioclast	99.3	2				
<b>Ha 9-5A</b>	70	2-phase L-V aqueous	Calcitized bioclast	135.2	1	16.7	3.51	d.l.	
<b>Ha 9-5A</b>	71	2-phase L-V aqueous	Calcitized bioclast	141.1	1	20.4	4	d.l.	
<b>Ha 9-5B</b>	30	2-phase L-V aqueous	Calcite cement	78	0.5	9.8	1.87	d.l.	
<b>Ha 9-5B</b>	31	2-phase L-V aqueous	Calcite cement	76.4	0.3	8.2	1.55	d.l.	
<b>Ha 9-5B</b>	32	2-phase L-V aqueous	Calcite cement	111	1				
<b>Ha 9-5B</b>	33	2-phase L-V aqueous	Calcite cement	95.5	1				
<b>Ha 9-5B</b>	34	2-phase L-V aqueous	Calcite cement	83.8	0.5	8.9	1.69	d.l.	
<b>Ha 9-5B</b>	35	2-phase L-V aqueous	Calcite cement	100.3	1				
<b>Ha 9-5B</b>	35b	2-phase L-V aqueous	Calcite cement	n.m.		9.3	1.77	d.l.	
<b>Ha 9-5C</b>	36	2-phase L-V oil	Calcitized bioclast	72.7	1				
<b>Ha 9-5C</b>	37	2-phase L-V oil	Calcitized bioclast	71.2	1				
<b>Ha 9-7A</b>	38	2-phase L-V oil	Calcitized bioclast	51.4	0.3				
<b>Ha 9-7A</b>	39	2-phase L-V aqueous	Calcitized bioclast	84.5	0.5				
<b>Ha 9-7A</b>	40	2-phase L-V aqueous	Calcitized bioclast	92	1				
<b>Ha 9-7A</b>	41	2-phase L-V aqueous	Calcitized bioclast	84	0.3				

Table 4. Synopsis of the Re-Os isotopic data of asphaltene fractions from oil from the Halahatang oilfield, Tarim basin, China.

Sample	Re (ppb)	±	Os (ppt)	±	<sup>192</sup> Os (ppt)	±	<sup>187</sup> Re/ <sup>188</sup> Os	±	<sup>187</sup> Os/ <sup>188</sup> Os	±	rho	*Os <sub>i285Ma</sub>
<b>Ha 9</b>	0.81	0.01	37.4	0.4	12.9	0.2	125.4	2.8	1.66	0.04	0.723	1.06
<b>Ha 9rpt</b>	1.23	0.02	57.2	0.7	19.5	0.4	125.2	3.2	1.74	0.04	0.732	1.14
<b>Ha11</b>	0.56	0.02	12.1	0.5	3.9	0.4	283.3	30.4	2.30	0.24	0.920	0.95
<b>Ha701</b>	0.07	0.01	4.9	0.3	1.7	0.2	78.7	16.0	1.48	0.19	0.612	1.11
<b>XK4-3</b>	0.85	0.02	8.8	0.6	2.3	0.5	736.8	147.9	4.68	0.93	0.989	1.17
<b>Ha15-2</b>	9.47	0.03	35.2	0.5	11.4	0.3	1655.2	39.7	2.25	0.05	0.970	-5.63
<b>Ha15-2rpt</b>	5.86	0.02	22.0	0.4	7.1	0.3	1636.7	59.9	2.25	0.08	0.983	-5.54

\*Os<sub>i285Ma</sub> = <sup>187</sup>Os/<sup>188</sup>Os measured calculated at the time of oil generation (258 Ma) to yield the initial <sup>187</sup>Os/<sup>188</sup>Os composition.